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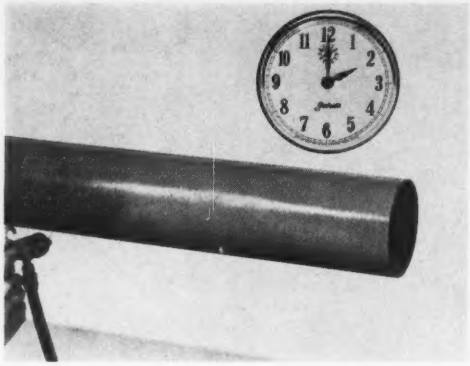

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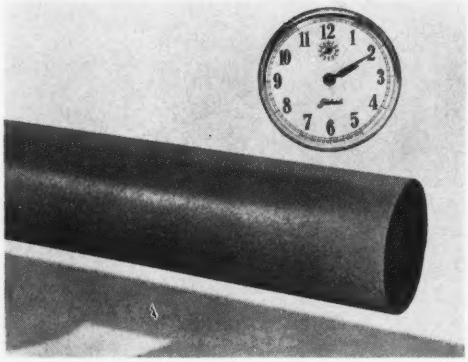

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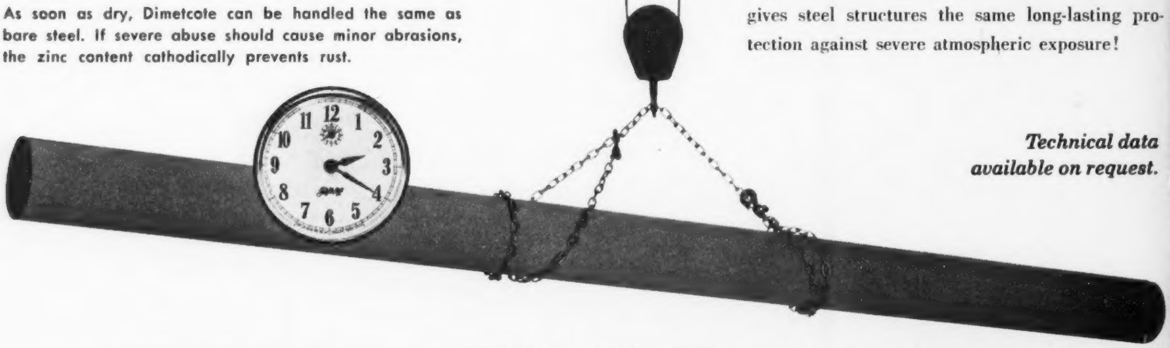

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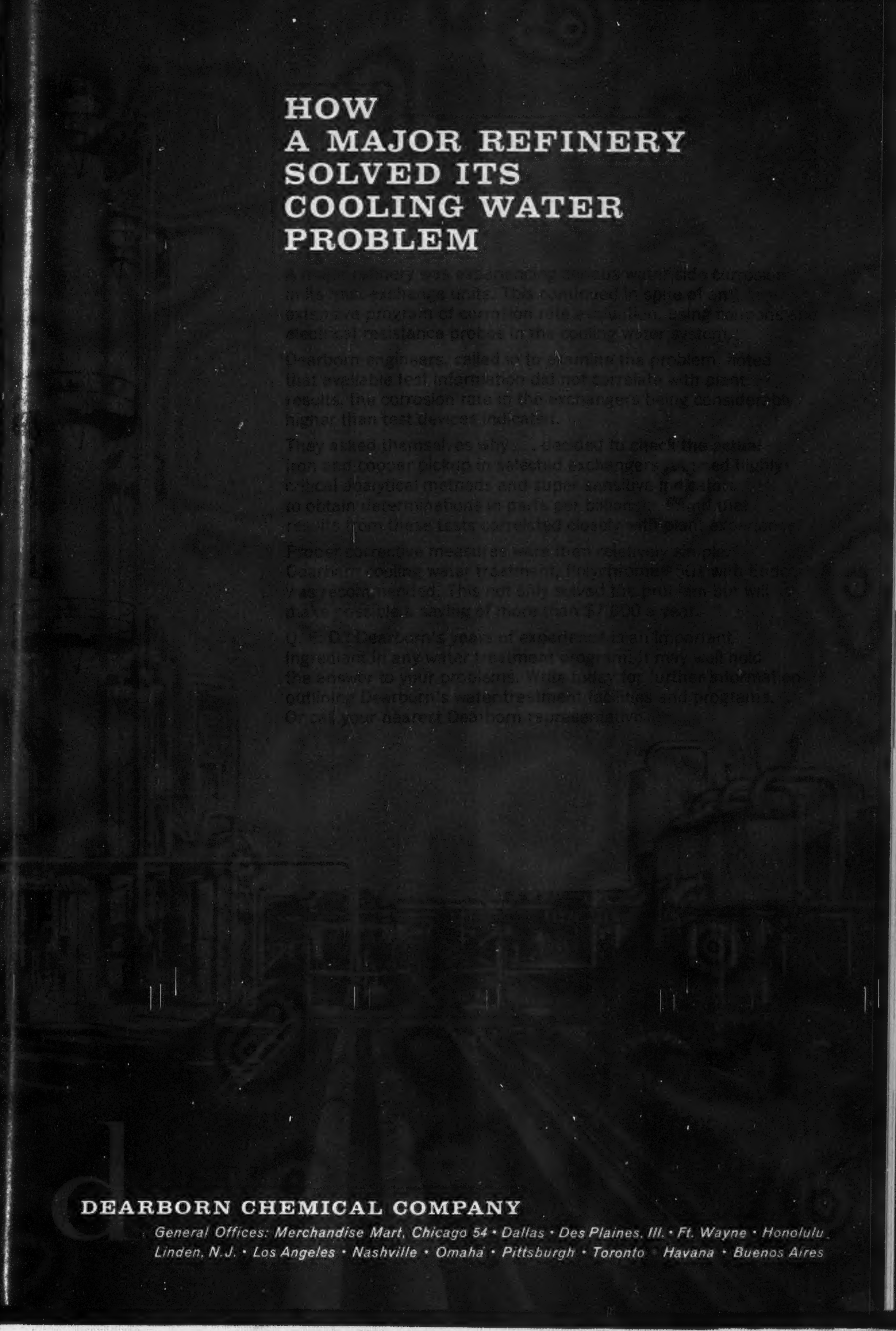

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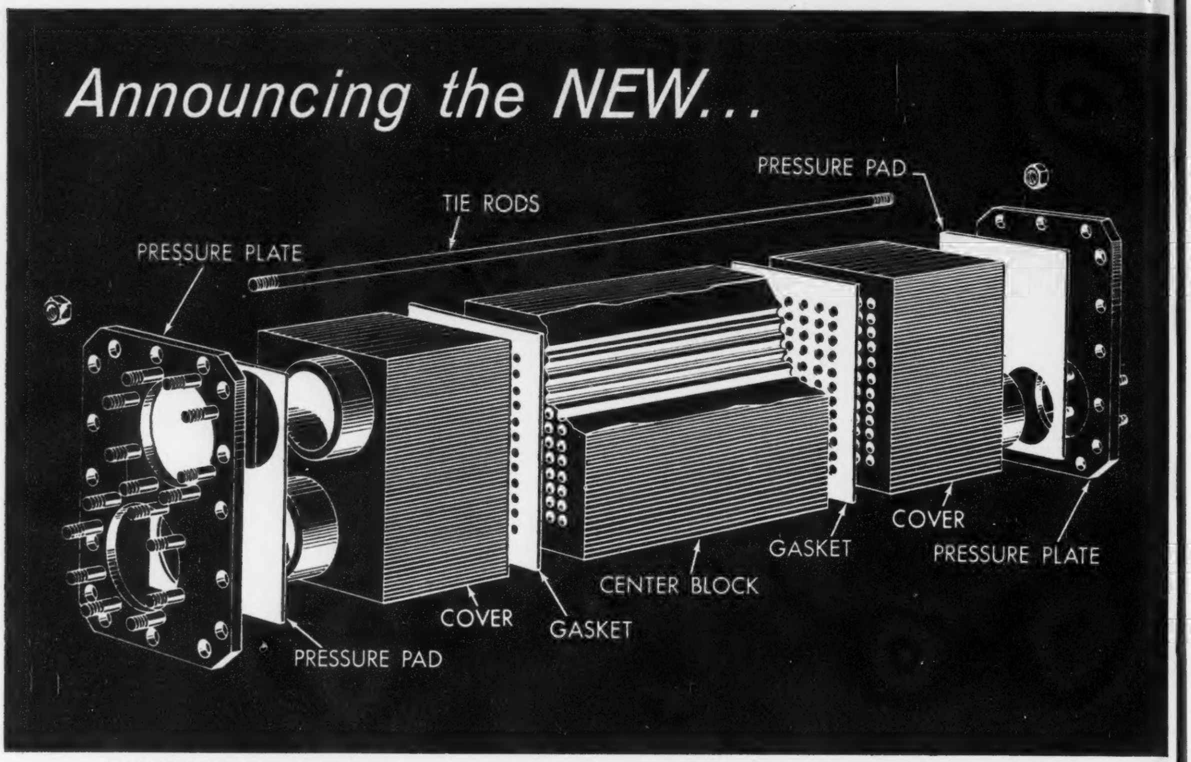

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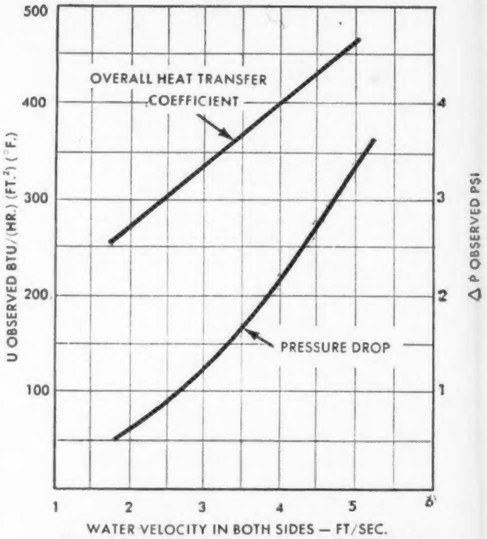

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This Month in Corrosion Control...

HYDROGEN FLUORIDE ALKYLATION process units are susceptible to attack by corrosives, but these attacks can be avoided or minimized by proper choice of materials and good housekeeping. Details of the tests leading to the introduction of this petroleum refining method are given, including initial laboratory and pilot plant experience. Reactions of various materials with the process streams are reported. Operators of plants using this process will find the data in this article useful in helping avoid corrosion damage. Turn to Page 9.

UTILITY POLE HARDWARE materials and coatings are discussed in an article beginning on Page 14. Comparisons are made among hot dip aluminum, zinc-filled coatings, sprayed metal and fluidized bed resin coatings. On the basis of performance and cost, galvanized coatings are preferred, the article concludes. For an opposing viewpoint, turn to Page 101.

OIL REFINERIES seldom, if ever, report the brittle failure of chromium-molybdenum steel. The first such report in more than 30 years was of a 1½-inch pipe which failed suddenly after seven years at 850 F, 400 psi. Examination showed segregated carbides believed to have been caused by local heating and severe hammering to dislodge coke caused embrittlement. Turn to Page 20.

METAL HIGHWAY structures are susceptible to many types of corrosion attack. Some of these are described and illustrations of the consequences given in a short article beginning on Page 22.

WIRE used in prestressed concrete were tested to determine to what extent hard drawn, stress relieved steel was superior to oil tempered strand and how calcium chlorides used in concrete corroded the wires. Three-year laboratory tests showed wires in concrete without calcium chloride suffered little or no loss in strength while those in concrete containing calcium chloride were badly corroded. Turn to Page 24.

BRIDGES ACROSS SAN FRANCISCO BAY consume 29,000 gallons of paint and cost over a million dollars a year for atmospheric corrosion prevention. Some interesting details of this vast maintenance program are given beginning on Page 26. Details are given about surface preparation, including formulation of cleaning compounds, paint systems, special problems and considerable cost data are revealed.

FUNDAMENTALS of corrosion control will be emphasized at the 3-day Greater Boston Section Corrosion Short Course to be held June 20-22 at Wentworth Institute, Boston. Detailed program information will be found on Page 49.

BLAST FURNACE SLAG, contrary to reports in the past, was found to be substantially inert versus steel when used as an aggregate in reinforced concrete. After three years prestressed concrete structures using the slag as aggregate were found to present every evidence of a long, trouble-free life. See Page 79.

ASPHALT MASTIC COATINGS designed for corrosion protection and/or weighting purposes on pipelines are critically examined in an article beginning on Page 81. Authored by the late Lyle R. Sheppard, the article concerns development of laboratory tests designed to provide data significant in estimating service life. Persons concerned with the application of coatings to pipelines will find useful information in this article and in the extensive discussions appended to it.

CORROSION PRODUCTS FROM METALS in steam condensate were examined with the view of determining the correlation of the temperature and composition of the condensate with corrosion products in an article beginning on Page 95. Metals examined were carbon steel, cast iron, admiralty metal, Muntz metal, aluminum 3S and zinc.

ALUMINUM and ALUMINIZED STEEL pole line hardware was found to perform better than galvanized hardware in 3-year marine atmosphere tests conducted in Hawaii. The article beginning on Page 101 also shows relationship between thickness and service life. For a contrasting view turn to Page 14.

ALUMINUM exposed to 300 C high purity water has a corrosion resistance related to the distribution of second phase particles in the alloy. Turn to Page 105.

VANADIUM COMPOUNDS will catastrophically corrode Type 310 stainless steel at 1900-2000 F under some conditions. A discussion of this effect, important to operators of high performance equipment burning residual fuels, begins on Page 109.

MILD STEEL submerged in harbor water up to 552 days can be protected from corrosion almost completely by cathodic protection. A study, which also gives corrosion rates on unprotected mild steel panels in harbor water begins on Page 112.

EIGHTEEN ALLOYS were tested in hot ammonia atmospheres over long periods to compare corrosion rates obtained in less aggressive ammonia environments. Nickel-base and iron-nickel-chromium systems were substantially more resistant to nitrogen than alloys richer in iron and chromium. Turn to Page 115.

ZIRCONIUM WELDMENTS exposed to hot hydrochloric acid were attacked in zones near welding as result of a precipitated phase in grain boundaries. Preventive methods are suggested in the article beginning on Page 120.

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How NACE Helps Train Corrosion Engineers

DURING the past eleven years the National Association of Corrosion Engineers has sponsored or participated in holding 82 courses designed to inform and train men interested in corrosion control. So far this year seven such courses have been held or scheduled and there probably will have been eight by the end of the year.

Because there is little time or opportunity for special attention to corrosion theory (and even less for corrosion control practice) in most formal engineering courses, the engineer whose assignment involves this specialty needs the time-saving help these courses provide. In recent years there has been an increasing tendency to restrict the subject matter for courses to specific subjects or specific industrial orientations. This is a natural outgrowth of increased specialization in the corrosion control field.

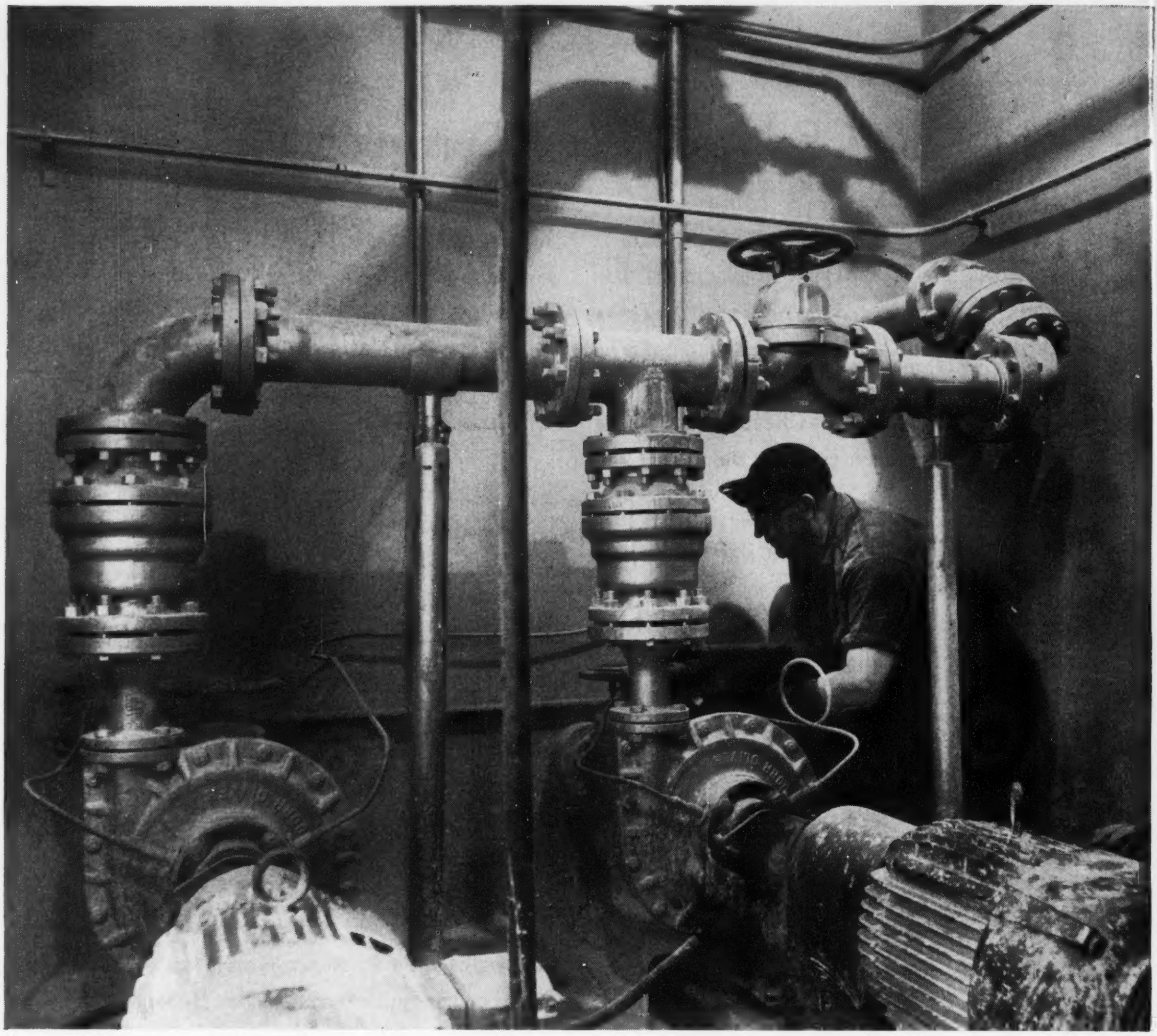
Nevertheless, a majority of the courses both in number and in numbers participating concern themselves with underground and underwater problems. This stems from the origin of NACE among pipeline and petroleum engineers and follows logically from the fact that a majority of NACE members are connected with the petroleum industry in one way or another. Some of the courses with the longest history are directed at operating personnel of pipeline and petroleum and gas processing companies.

NACE expects to increase its activity in the field of corrosion education. NACE plans to continue sponsoring courses, to cooperate with others holding courses and to seek new areas where courses are needed. This activity certainly holds the promise of being a major contribution to companies trying to solve their corrosion problems. This contribution is completely consistent with the aims of the association.

A discussion of the corrosion control short course program would be incomplete if due credit were not given to the NACE members who transmit their knowledge, contribute their time and often their money to this activity. Credit is due also to the companies that employ these men and make their participation in the courses possible by giving them the time, travel money and encouragement to instruct others who need to know about corrosion control.

There is some reason to hope that more short courses of the future will be organized and held for engineers in industries other than petroleum. More courses are needed such as the one held in September, 1960, at Ohio State University on process industry corrosion problems, or the one held last May by NACE's Niagara Frontier Section on organic coatings.

Anyone who believes there is an opportunity to hold a short course and who needs advice and assistance may call on NACE. The association will be glad to do everything it can to help. Properly organized and staffed courses have proved to be successful and fruitful in the past; there is every reason to believe they will continue to be in the future.



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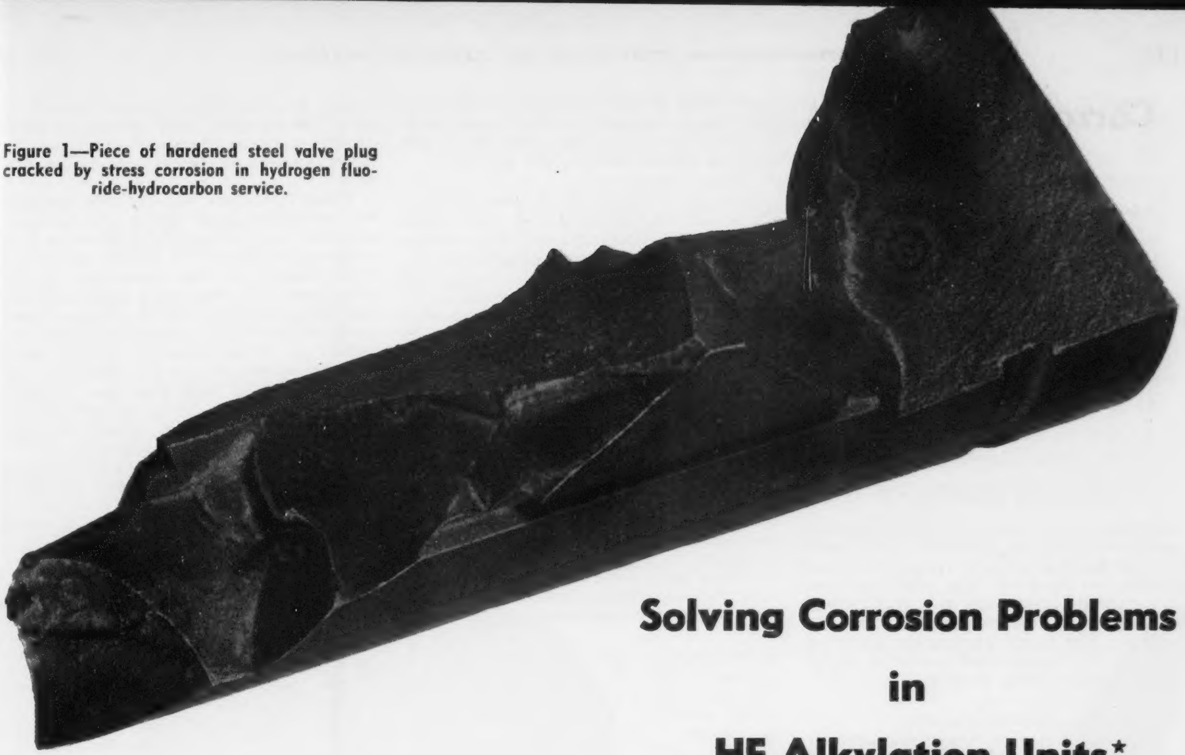

Figure 1—Piece of hardened steel valve plug cracked by stress corrosion in hydrogen fluoride-hydrocarbon service.

Solving Corrosion Problems in HF Alkylation Units*

TECHNICAL TOPICS

D. J. Bergman and G. W. G. McDonald
Universal Oil Products Company
Des Plaines, Illinois

Introduction

THE STORY of the development of the Universal Oil Products Company's hydrogen fluoride alkylation process from the laboratory to full scale commercial production of aviation gasoline component furnishes a background of interesting information and guide for operators of today's alkylation units. A brief review of the metallurgy is indicated.

It should be remembered that development progressed from a laboratory process to a pilot plant which would fit on top of an ordinary office desk and then to 23 units, ranging in capacity from 640 to 3840 B/D of alkylate. All were designed and under construction before the first commercial unit went into operation.

Experience in handling anhydrous hydrogen fluoride in process units was non-existent when these tests began. Information on equipment and corrosion rates was available only from acid manufacturers and from laboratory data. Further experimental data were obtained from a test circuit which consisted of a centrifugal pump, some piping joints and various types of valves. Prior to this, almost all transferring of acid by the manufacturers employed a blowcase—an intermittent operation which discharged air heavily laden with acid fumes into the atmosphere.

A considerable amount of corrosion data for anhydrous HF and for the HF-water azeotrope, familiarly known as "constant boiling mixture" (CBM), was available from laboratory tests. This information pinpointed obvious and speedy attack, but the short-term tests did not indicate potential stress-corrosion problems or the corrosive properties of various mixtures with other compounds, particularly those containing sulfur.

Metallurgical Problem Is Explored

Corrosion data in the literature say that lead, which is highly resistant to corrosion in the manufacture of sulfuric acid by the lead chamber process, and also to aqueous HF has very poor resistance to anhydrous HF. On the other hand, silver and Type 316 stainless steel show high resistance, exceeded only by Hastelloy C. Hastelloy B, closely related to Hastelloy C, is shown to have a fairly high average corrosion rate and also was perforated locally. Carbon steel, copper and bronze all appeared to be about on a par. However, a critical look at this information in the light of the operation of the early plants emphasizes the significance of data derived from operations under plant conditions as compared to that from laboratory work.

Because HF ionizes over a very wide range of dilution with water, copper or bronze exposed to HF will be rapidly corroded when connected to steel. Accordingly, it was recognized early that steel tubes in exchangers or coolers were proper in acid service. Because during the primary development stage, seamless steel tubes were in short supply, many exchangers in acid service were equipped with longitudinal-seam welded tubes, some of which were of quality which made them useful only for mechanical tubing. In some cases after short service it was possible to see through the longitudinal weld at regular intervals—literally as if the seam had been stitched with wire.

Corrosion data cited the very poor resistance of cast iron to HF, which, therefore, was eliminated from consideration. Fortunately, cast steel showed good resistance and was used successfully in valve bodies, pump cases, etc.

Silver's Indicated Resistance Highest

Laboratory data on silver showed it had the highest resistance to the constant boiling mixture of HF and water of any metal tested. Consequently, acid regenerators in the first plants were built of steel clad with silver approximately 1 mm thick. Not until the plants were in operation was it discovered that anhydrous HF exposed to hydrocarbons containing only traces of sulfur would absorb all of the sulfur. The sulfur in the acid then attacked the silver, converting the lining to silver sulfide and thus exposing the steel to attack by aqueous HF.

Another laboratory experience ruled against the use of Monel in the early plants. A Monel beaker had been used to contain an acid-water mixture covered with hydrocarbons. After standing over-night it was emptied and decontaminated. When it was noted that the top of the beaker was somewhat oval, a slight pressure of the fingers was applied to make it round again. With a tinkling sound a crack ran all around the beaker on a line where the interface had been. Accordingly, it seemed obvi-

(Continued on Page 10)

★Revision of a paper titled "Corrosion Experience With HF Alkylation Units," presented at a meeting of Canadian Region, Eastern Division, National Association of Corrosion Engineers, Toronto, Ont., January 18-21, 1960.

Corrosion Failures on Bolts and Valves

(Continued From Page 9)

ous that even though Monel showed good resistance to corrosion, it was subject to serious stress corrosion and should not be used. Not until the first commercial plants were in operation was it found that this experience was misleading. The crack apparently occurred because the beaker had not been stress relieved after deep drawing.

Valves Were Serious Problem

Perhaps the most serious metallurgical problem in the original plants involved plug valves used for tight shutoff. Because HF was considered a hazardous chemical, it was deemed imperative to use a minimum number of flanges and connections; where valves were required, grease-sealed cocks were used.

These cocks quickly developed problems in service. The plugs, made of steel with approximately 0.45 percent carbon, had been hardened to 500 Brinell or more by heating and quenching. Quenching the large plug in a tub of water caused shrinkage of the outside skin while the inside was still hot. After cooling to a uniform temperature the outside was under relatively high compression and any shrinkage cracks which formed usually were invisible. However, these cracks furnished the starting points for stress corrosion attack, as shown in Figure 1. Even without cracks the highly stressed surface of the hardened plug was extremely vulnerable. Consequently, although a plug in hydrocarbon acid service might be in excellent working condition when the valve was closed following startup, the cock might be found completely inoperable a month later when it was necessary to operate the plug to isolate a piece of equipment. In some cases the plug came out of the body in pieces, almost like gravel.

The use of Monel-lined non-hardened plugs with Stellite-lined bodies as a field replacement eliminated the plug cracking and still gave the highly desirable difference in hardness between plug and body for good plug cock operation.

The lubricant in the grease-sealed cocks brought another problem. It tended to foul the lines and migrate around the plant when employed in quantities sufficient to assure a seal.

Hundreds of laboratory tests had been made to find suitable materials for gaskets, valve packing and pump stuffing boxes. Asbestos disappeared in a short time. Aluminum foil was attacked but seemed to give reasonable life. Carbon in some cases appeared to be quite satisfactory, but in others it would disintegrate in air after exposure to HF. The first units were equipped with ring-type flanged joints. Carbon and Stellite seals were used on pumps.

Instrumentation Was Difficult

One of the early design problems was that acid liquid levels had to be determined solely by mechanical instruments, because gage glasses could not resist the corrosive effect of HF. The sticky corrosion product film which formed on steel and stainless steel instrument parts tended to freeze them and, consequently, determination of level was unsatisfactory.

Abstract

Presents review for guidance of present-day operators of anhydrous hydrogen fluoride alkylation process. Covers early laboratory and pilot plant tests and metallurgical problems encountered in first commercial units. Steel was recognized early as a material for construction of principal components of process units but was not satisfactory for all services. Monel, ruled out initially, later was found satisfactory for service as trim and in various kinds of valves. Valves with steel cocks failed when the HF attacked them, often sealing them into inoperable condition. Grease used for lubrication was troublesome in the stream.

Numerous kinds of packing were tried, but none were entirely satisfactory at first. Monel and fluorocarbons made available later solved packing and valve-seat problems and also problem of protecting glass sight level gauges.

Steel bolts failed rapidly from stress corrosion cracking when small leaks resulted in acid attack. K-Monel bolts also failed. Duranickel bolts were satisfactory.

Dangers of contamination of process stream by water or water by acid are cited. Constant pH checks are advocated as a control measure. External corrosion is small when good housekeeping is maintained. 8.4.3

Plant operators avoided this problem by providing gage cocks. An operator standing on the windward side of a gage column could open successive valves and note changes in the appearance of the discharge from the valve above and the one below the liquid level. Because gage cocks would stick occasionally, even two valves in series were not always satisfactory protection for the operator. Although it was vital that the operator should know the level of acid in the tank, or in some other vessel, the open gage cocks were a hazard.

The desirability of stress relieving welds in vessels and piping in alkylation service was recognized early in the design stage. Consequently, little stress cracking of steel was encountered.

Problems With Connections

Another problem did show up, however. Even though pressure testing had been carefully done and vessels and piping were completely leak free, small slag inclusions which might form a continuous path through a weld were quickly attacked by the acid and resulted in leaks. In many cases these could be blocked by peening, but frequently cutting out welds and rewelding was required.

Seal welding of screwed pipe connections also caused leaks. In the majority of cases these screwed connections had been made up either with pipe dope or retained enough oil from the thread cutting or makeup operation to cause porosity of the weld. (A similar problem arises when tube rolls are seal welded.) Elimination of oil in such connections is imperative if they are to be seal welded.

Modern techniques in welding and improved electrode composition have eliminated almost entirely the slag inclusion problem.

Flange Bolt Failure

Valve bonnets or flange connections developed another interesting metallurgical failure. When a small amount of acid leaked from a flange because of line strains or a pinched gasket, it would pick up moisture from the air to produce a weak solution on the surface of the flange bolts. This caused bolt failure from stress corrosion.

American Society for Testing Materials specifications on high tensile steel bolting materials for high temperature service are based on three minimum tempering ranges—1000, 1100 and 1200 F. Bolting material is usually furnished with 1100 F temper or less in order to assure high tensile strength. It seems quite possible that bolts tempered at 1200 F would have much greater resistance to stress corrosion even though the maximum tensile stress would be reduced considerably. The difficulty would be in making sure that bolts of this specification were properly identified and used only for the purpose intended, and that standard high tensile bolts were not accidentally substituted. An expedient is to use low carbon steel bolts.

Monel Bolts Also Cracked

Monel bolts also were used in valve

(Continued on Page 12)

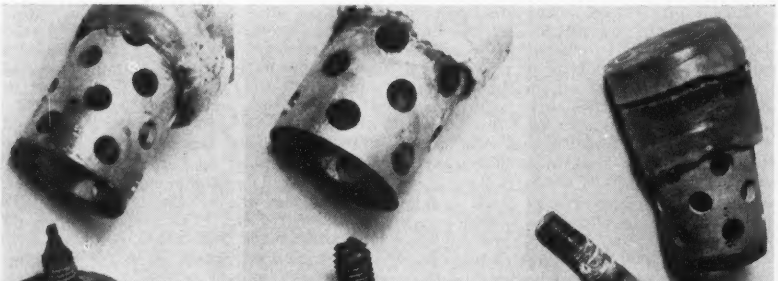

Figure 2—Effect of 1 percent hydrogen fluoride on carbon steel (left) K-Monel (center) and Duranickel (right) bolts.

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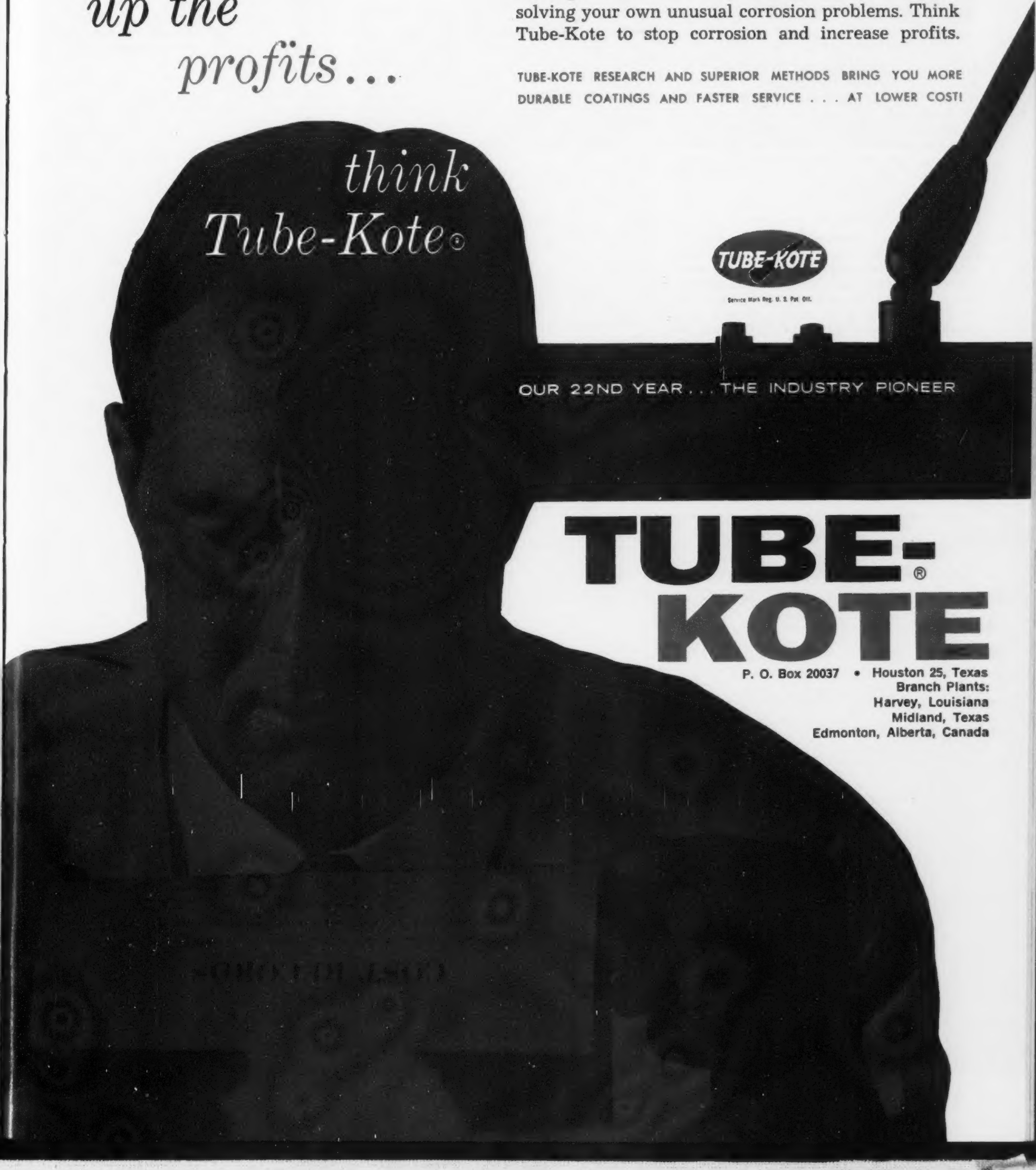
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Fluorocarbon Plastics Help Solve HF Plant Corrosion

(Continued From Page 10)

body and flange connections but they, too, were subject to cracking. This occurred because Monel bolts are made of K-Monel, in which a small amount of aluminum is added to obtain a solution-hardening alloy. Upon proper heat treating the strength of these K-Monel bolts, and especially the elastic limit, is greatly increased. At the same time, however, susceptibility to stress corrosion also is increased.

Duranickel(1) bolts show very much better resistance to stress corrosion than steel or Monel although they cost considerably more. This resistance is shown (in Figure 2) by a comparative test in which high tensile steel, Monel, and Duranickel were partially immersed in 1 percent acid solution while under load. In this test a 4140 bolt broke in just under six months and was very badly corroded. A K-Monel bolt lasted nearly seven months, cracking with very little visible corrosion. The test on the Duranickel bolt was discontinued after 11½ months. The bolt had not broken but surface corrosion was quite evident.

One serious source of corrosion in the original plants developed as a result of using bauxite to eliminate traces of HF and combined fluorides from alkylate or LP-gas streams. The acid combined with silicon compounds in the bauxite to form silicon tetrafluoride and water. This combination was much more corrosive than HF and caused numerous failures in equipment downstream from the bauxite towers. Substitution of distillation for bauxite towers in the new plants to remove HF and combined fluorides has greatly reduced corrosion problems.

Modern HF Plants

In modern HF alkylation plants, a typical flow diagram for which is shown in Figure 3, one of the important factors in the design is simplification and reduction of the number of connections. Use of valves with Monel trim has eliminated the freezing which occurred with steel valves and trim and cracking of hardened stainless steel trim. However, straight Monel was subject to galling and scoring. Grease-sealed Monel globe valves were the first really satisfactory valve in HF service, but still were susceptible to galling and scoring and, furthermore, the necessity of lubrication added to maintenance. Grease-sealed Monel gate valve also proved fairly satisfactory in large sizes.

Development of the fluorocarbon plastics in recent years has provided an excellent solution to the problems raised by the high reactivity of HF. These make ideal substitutes for asbestos as gasketing and packing material and have made possible an effective non-leaking joint. Polychlorotrifluoroethylene(2) sheeting has filled the need for some form of visible continuous level indication. Use of "through-view" gage glasses which have the glass separated from the acid by fluorocarbon has been very satisfactory except in a few instances where the plastic was damaged mechanically. This also has eliminated the potential safety hazard which resulted from various expedients, such as trylines, tryline probes, Monel trimmed magnet-bearing floats, and Monel torque-tube type level indicators and radiation type indicators.

The first really satisfactory reasonably priced valve, particularly in the small sizes, was the fluorocarbon seated globe valve. When the seat was prevented from flowing, this type of valve gave a positive shutoff and its softness reduced the damage often suffered by hard seats from foreign particles. Fortunately, polytetrafluoroethylene(3) is completely satisfactory in HF service.

An important factor in valve and piping safety is the use of HF-sensitive paint on flanges and bolts where acid leaks might cause failure. Change of color from orange to yellow—indicating a leak—calls for complete bolt replacement.

The best leak proof gasket is none too good for flanges in an HF plant. Spiral wound Monel and polytetrafluoroethylene gaskets have given excellent service within rated temperature limits. Monel steel rings also have been satisfactory.

Exchanger Problems

In the product fractionation section of an alkylation unit steel tubes still are recommended and stress relieving of tubes is required when U-tube bundles are used. Where a bad water condition is encountered 80-20 cupro-nickel is recommended because Admiralty tubing frequently is dezincified in a fairly short time. Floating head bolts occasionally

---

(1) Nominal composition (percent): Ni (plus Co), 93.90; C, 0.15; Mn, 0125; Fe, 0.15; S, 0.005; Si, 0.55; Cu, 0.05; Al, 4.50 and Ti, 0.45. Source: "Properties of Some Metals and Alloys," International Nickel Company, Inc., New York (August, 1959).

(2) Kel-F, produced by Minnesota Mining and Mfg. Co., St. Paul, Minn.

(3) Teflon, produced by Du Pont de Nemours & Co., Wilmington, Del.

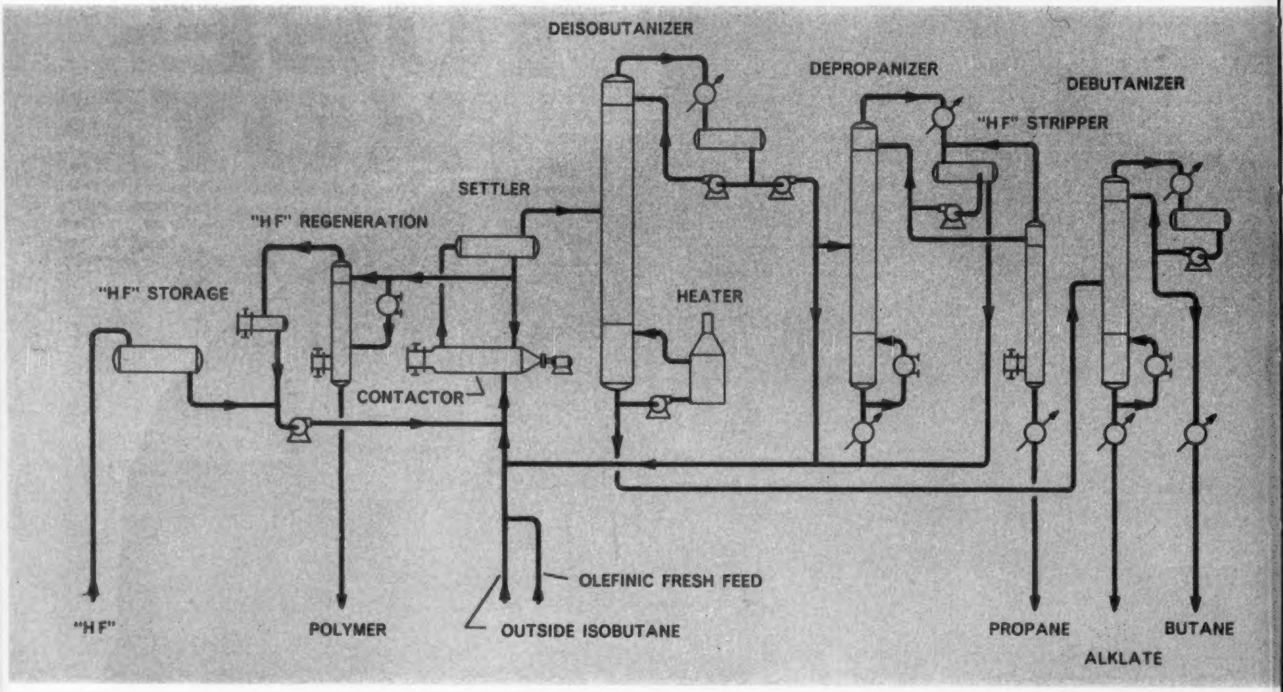


Figure 3—Schematic flow diagram of hydrogen fluoride alkylation.

fail because of stress corrosion cracking or embrittlement when subjected to an acid environment. Either Duranickel bolts or low carbon steel bolts will eliminate this problem.

It is fortunate that good carbon steel is one of the most satisfactory materials of construction in HF service. Complete stress relieving of vessels exposed to acid is specified after fabrication in order to eliminate possible stress corrosion. Minor repairs have been made to such vessels without subsequent stress relieving using 3/32 or ⅛ inch electrodes, low current density and taking care not to overheat the work.

The surface film of corrosion products on steel is very adherent and, consequently, close fitting assemblies usually are frozen together. In order to disassemble instruments, wear rings, mechanical seals, etc., annealed Monel trim is imperative.

Good Housekeeping Limits Exterior Corrosion

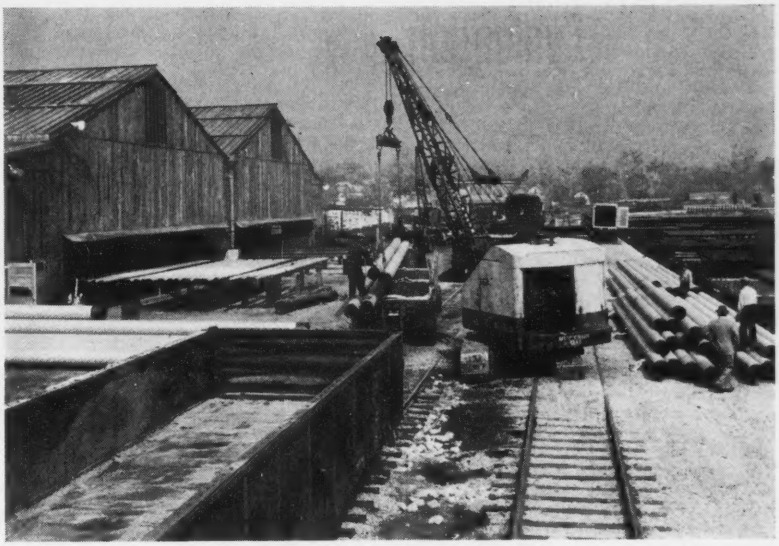
With good operation and good housekeeping exterior corrosion is no problem around HF units. Where plant upsets result in discharge of HF to the air, damage to buildings and window glass may result. Also, corrugated steel roofing and other steel generally show heavy rusting. A really bad upset may be caused by leakage of HF into plant cooling water. Because this occurs occasionally, keeping a pH recorder in the circulating cooling water system is recommended as a precaution against serious and widespread corrosion from this source.

Contamination of acid with water causes corrosion and also results in loss of acid through the regeneration system which rejects a constant boiling mixture. Therefore, it is common practice to use oil instead of water for pressure testing of acid alkylation equipment. If this is impractical, the unit should be dried thoroughly with gas before introducing HF. Hydrocarbon feed streams likewise should be thoroughly dried and careful attention paid to the operation of desiccant driers or distillation tower drying so that no water is carried into the acid section of the plant. Similarly, acid regenerator facilities should be maintained in good condition and operated to maintain high acid concentration, otherwise both plant and product quality will suffer.

Cooling water system pressure seldom is higher than process side pressure so leakage of water into the stream from this source rarely occurs. However, leakage from a steam heated exchanger could occur when the steam pressure is higher than the process side pressure. Conversely, leakage of acid or hydrocarbons into a steam system is hazardous because of unexpected corrosion. This may be readily detected by periodically testing condensate with a pH meter.

Conclusions

Major problems of both plant metallurgy and operation were solved in advance in laboratory and pilot-model tests. Current operation problems most frequently arise from complacency and the ease with which past lessons can be forgotten with the passage of time. Constant vigilance is necessary to guarantee continued high standards of housekeeping and maintenance. This is by far the best and cheapest way to prevent corrosion problems from developing in an alkylation unit.



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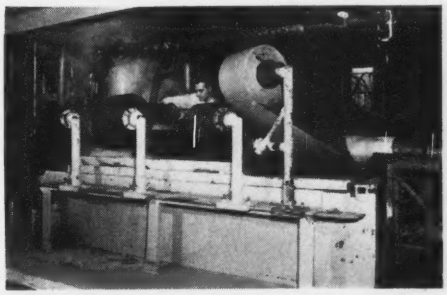

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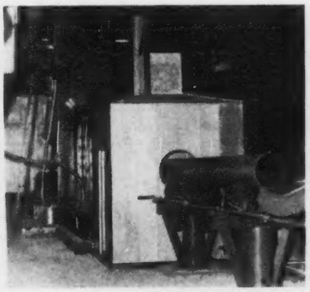

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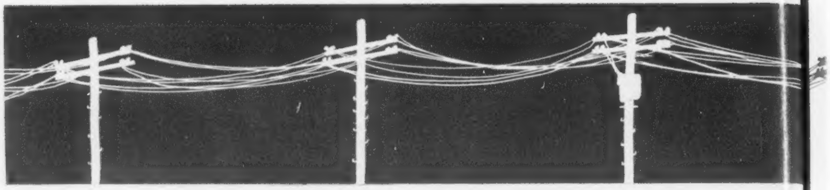
Evaluation of Some Materials and Coatings for Utility Pole Line Hardware*



Introduction

THE COST of replacing corrosion damaged line hardware is of growing concern to the power and communication industries. Faced with high labor costs coupled with a need for expanded overhead plant to meet increased service demands, the utilities are looking for longer useful life from their pole structures. The goal is to match hardware life to the already proven pole life, thereby eliminating rework of overhead structures.

Both utilities and manufacturers in the industry are examining new corrosion resistant materials and coatings, seeking extended hardware life. While a number of sources have reported corrosion resistance tests that show encouraging results, hardware products fabricated from corrosion resistant metals or protected by new coatings are available only to a limited extent. No full measure of success has been achieved in meeting the corrosion problem.

Purpose of this article is to examine some of the factors which dictate the manufacturers' choice of hardware materials and coatings and their acceptance by the utility industry. Influence of these factors on new materials will be shown and a variety of the developments will be compared to hot dipped galvanized steel.

Line construction hardware must fulfill three basic requirements: function, durability and reasonable cost. To build a hardware product which will meet these requirements, the manufacturer must realize that the simple and commonplace nature of individual hardware items belies the complexity associated with the full line of products needed to build today's overhead plant. Selection criteria for corrosion resistant material differ from those applying to pro-

Figure 1—Strap test specimens and lag screws. Specimen on left is galvanized; one on right is hot dip aluminum coated.

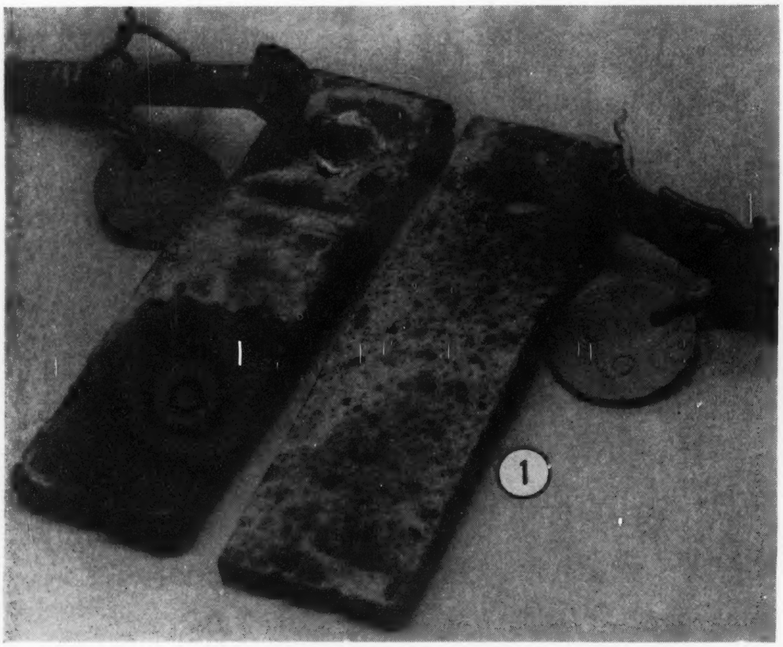
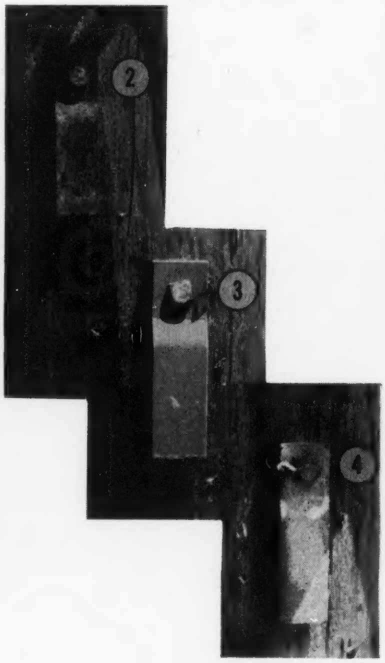


Figure 2—Hot dip aluminum coated strap test specimen. Strap test specimen covered with zinc filled coating is shown in Figure 3. Figure 4 is a strap specimen of hot dip galvanized. All three specimens were exposed for three years at a Bermuda coastal test area.



★ Revision of a paper titled "Experiences in Evaluating Protective Materials for Utility Hardware" presented at the North Central Region Conference, National Association of Corrosion Engineers, October 19-20, 1960, Milwaukee, Wis.

Abstract

Examines some factors dictating manufacturers' choice of hardware materials and coatings for utility pole hardware. These factors are corrosion resistance, cost, adaptability and compatibility. Discusses use of aluminum, stainless steel and high strength, low alloy steels as materials for corrosion resistance. Also discusses corrosion resistant coatings: hot dip aluminum on steel, zinc filled coatings, spray metal coatings and fluidized bed process. Comparisons are made of cost of materials and coatings.

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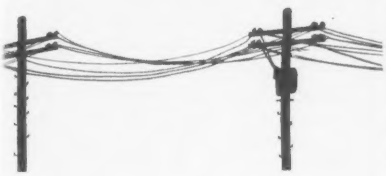


John S. Melvin

Line Material Industries
South Milwaukee, Wisconsin

tective coatings for steel. However, both systems involve the following considerations:

Four Points of Consideration

1. Universal Corrosion Resistance

With galvanized steel as a benchmark, a new coating or material system must show superior corrosion resistance to all atmospheres and soil conditions. Requirements of economy in material procurement, tooling and process facilities make one universal system extremely desirable. The limited availability of corrosion resistant hardware today can be attributed to the failure of currently used materials to economically provide this universal corrosion resistance.

2. Cost

Justification for adopting a new corrosion resistant hardware system must be based upon savings in replacement labor and material. Ideally, the cost should be equal to or less than that of galvanized steel although a nominal premium could possibly be tolerated. The premium will vary among operating utilities and can be determined only by detailed economic study. However, a 25 percent premium over galvanized steel has been accepted as reasonable by several leading utility companies.

3. Adaptability

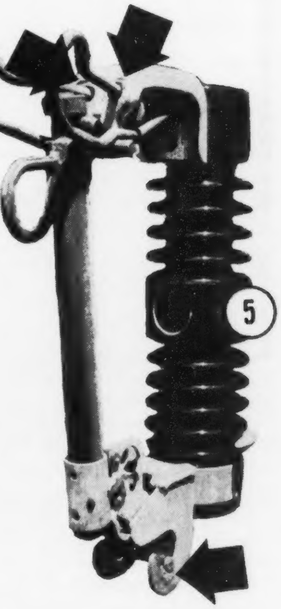
Pole structures require a wide variety of parts to fulfill application needs. All parts must be equally corrosion resistant if they are to parallel the useful life of a structure. Therefore, the new system must be adaptable to all common hardware. A weak link requiring early replacement will offset labor savings anticipated in constructing the balance of the structure with corrosion resistant hardware.

4. Compatibility

Line hardware must be compatible with materials used commonly in overhead plant construction. The factor of galvanic corrosion from contact with copper, aluminum cable and galvanized steel must be considered. Chemical attack by pole preservatives is a somewhat remote possibility but can present problems.

A prospective hardware material also must meet these supplementary requirements: (1) Be available in shapes and sizes needed for a full line of hardware products. (2) Be workable with commonly available fabrication methods and facilities. (3) Be relatively free from discrete forms of mechanical and corrosion problems.

Figure 5—Fuse cutout assembly. Steel parts were covered with a zinc filled coating. Exposure was for two years at Bermuda coastal test area. Arrows indicate where zinc filled coating deteriorated.



Materials Tested For Pole Line Hardware

Aluminum

Aluminum has been used to a limited extent in line hardware products for several years. For universal application, however, it fails to satisfy most requirements. Aluminum does not have sufficient corrosion resistance because it is soluble in many acids and alkalies. It is costly, both as a basic material and in processing when compared to mild steel. A serious problem is its lack of compatibility with other metals, particularly copper.

Stainless Steel

From the standpoint of mechanical and physical properties, stainless steel would perform very well as a pole line hardware material. Its high cost limits its use, however, to applications where severe environmental conditions justify this cost.

High Strength-Low Alloy Steels

Periodically, consideration has been given to the use of high strength-low alloy steels. This type material, because of benefits of the alloying elements, possesses much better mechanical properties and considerably better corrosion resistance than structural carbon steel. For applications where these properties are the controlling factor, high strength-low alloy steels can be used to good advantage. However, availability of this material in shapes and sizes to fill all hardware requirements is poor. This means that, while basic products such as sheet, plate, angles, rounds, etc., are available (in mill shipments not warehouse), items such as bolts, nuts and washers are not stocked in all necessary sizes. Cost of these as specialties prohibits considering their use in pole line hardware. From standpoint of adaptability, therefore, the high strength-low alloy steels do not satisfy the evaluating criterion.

High strength-low alloy steels also create many problems and corresponding higher costs in manufacturing processes. Tools used for machining, stamping and forming these materials have only half the life of those used for mild steel, with accompanying high die maintenance costs. Piercing, shearing and forming tonnages are approximately twice those required for mild steel. High strength-low alloy steels work-harden faster when subjected to cold forming, thereby limiting the amount of forming that can be done without process annealing.

Keeping these special steel stocks separate from mild steel as raw material and as in process inventory at the manufacturing plant is troublesome. A similar problem faces any utility using galvanized high strength-low alloy steel hardware.

In service, the zinc coating will be removed at the same rate, and the first stages of rusting will appear at about the same time as with galvanized mild steel. It is, therefore, necessary to keep special inventory and record all points where it is used in order to benefit from the superior qualities of the base metal. The additional cost of these operations plus the much higher initial investment place high strength-low alloy steels above the cost limit as a universal material for hardware products.

Copper bearing steels (0.20 percent minimum copper) are in much the same position as high strength-low alloy steels. They show high resistance to corrosion but have limited availability. Use of these steels would increase the cost of pole hardware products considerably. If used only partially on a structure, this increased outlay would not be beneficial.

Coatings Tested and Estimate of Their Suitability

A coating to be considered for use on hardware products must not require processing injurious to the properties of the base metal and must be reasonably available through commercial outlets.

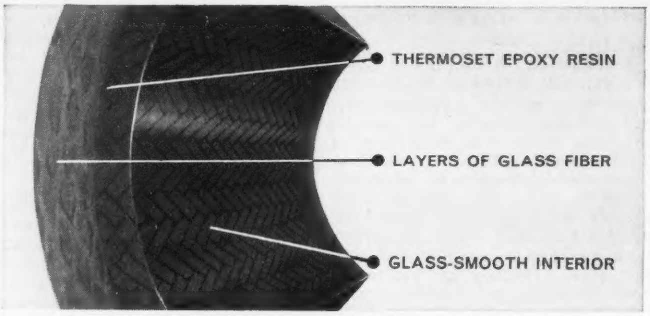
Hot Dip Aluminum on Steel

In early stages of development, the aluminum hot dip process was thought to provide the answer for an economical, long-life protective coating. Published information and experimental test data indicated that the corrosion resistance of this type coating was considerably better than that of galvanized coatings. Further testing confirmed the initial data.

Figure 1 illustrates the superiority of aluminum coatings in comparative tests exposed under very severe conditions. These specimens were attached to a pole by lag screws and exposed four years at a Pacific Coast site where they were subjected to periodic sprays from the ocean and drying from the sun and offsea breezes.

The aluminum coated lag screw was severely attacked in the area exposed

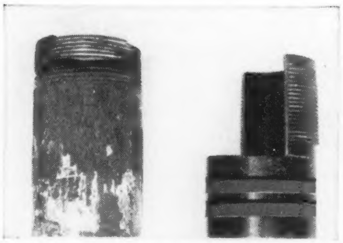
(Continued on Page 18)



EPOXY RESIN AND GLASS FIBERS combined in a high-speed centrifugal casting operation. High quality components processed under rigid quality controls bring to industry a unique and efficient non-metallic pipe.

"Impossible" corrosion, temperature and pressure problems solved by Fibercast® epoxy pipe

- Operating temperature range wider than any non-metallic pipe (–65° to 300° F.)
- Operating pressure range extends to 1000 psi.
- Effectively combats corrosion, contamination, abrasion, scaling, electrolytic action.



Fibercast Tubing (right) used to suspend a 1,200 lb. pump for 3 years in a salt water supply well. *There was no loss in strength.* The damaged plastic-coated steel nipple (left) was used in the same installation, failed after 3 months' service.

WHAT is Fibercast?

It is a centrifugally cast, thermoset, epoxy resin reinforced pipe that handles temperature and pressure problems where no other non-metallic pipe will do.

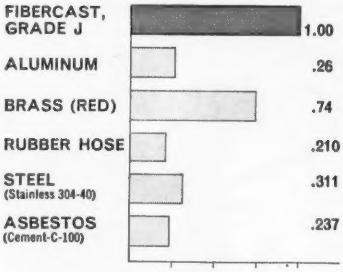
Its body of woven glass fibers provides resistance to high tension forces. These fibers, combined by adhesion, are imbedded and bonded by heat in epoxy resin. The result is a strong, long-lasting pipe with remarkable ability to withstand high pressure and temperature in corrosive environments.

WHY use Fibercast?

For a multitude of reasons. Because its advantages range from superior resistance to heat, pressure and corrosion, to its ease of handling, light weight, dielectric properties and structural stability. Because Fibercast's long service life alone would justify choosing it over other materials. Because even more expensive metal pipe or pipe with thermo-plastic interior coatings cannot match Fibercast's proven durability. Case histories and accurate testing have proved over and over that Fibercast performs better, lasts longer, costs less. Out of 338 common corrosive solutions, Fibercast competently handles 320. And naturally, Fibercast's unique and lasting qualities under such conditions mean that it drastically reduces maintenance and replacement costs, too.

Although Fibercast's light weight makes it correspondingly easy to work with (it is less than 25% the weight of steel), it has the same basic linear coefficient of expansion as steel (7.06 x 10^{-6} to 8.25 x 10^{-6} in./in./°F.). Fibercast's strength permits installation on pipe racks with span lengths generally used for metal pipe.

COMPARATIVE LIFE DATA*



**Basing Fibercast as unit life of 1 and others as comparative percentages thereof.*

The pipe has a smooth interior, with a Hazen-Williams C Flow Factor of 147. This cuts friction losses, aids flow and tends to resist build-up. It is a non-conductor (accepted by the electrical industry as a superior insulator), and is not subject to cold flow. A low coefficient of heat transfer (3.0 x 10-3 Cal/CM²/sec/CM°C.), minimizes heat loss and may even eliminate heat tracing.

All three major systems of joining all pipe are used with equal success on Fibercast. They are: standard flanged, cemented, and threaded and coupled with a complete selection of Fibercast fittings (the world's largest line of corrosion-resistant epoxy pipe fittings).

Safety, too, is an important reason for using Fibercast Pipe in transporting dangerous chemicals, particularly in areas where the pipe is adjacent to personnel. Most piping materials corrode undetectably from inside-to-out and can suddenly burst without warning, inflicting injury to personnel. Because of Fibercast's woven glass fiber construction, such occurrences, if they ever occur, *are* detectable in minute, repairable leaks.

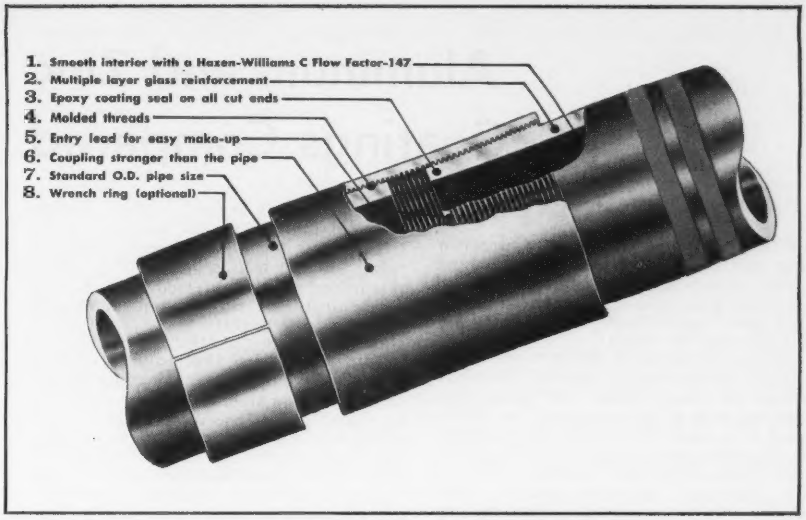
WHO uses Fibercast?

The petroleum industry . . . chemical . . . petro-chemical . . . nuclear energy . . . textile . . . paper . . . and food-processing industries . . . countless operations handling acids, alkalies, salt water and other corrosive liquids under pressure can all use Fibercast profitably.

In oil country, Fibercast is already widely acclaimed for superior performance in rugged, abusive and even high pressure installations like salt water disposal wells and horizontal lines. In cases where other pipe and tubing required replacement after just a few weeks of service, Fibercast is still performing efficiently after many years.

Workman is painting a section of Fibercast Pipe bearing 32% HCl at Shell's Texas Chemical plant. Note that Fibercast has sufficient strength for installation on span racks with the spacing normally used for metal pipes.

In petro-chemical and chemical-processing plants, Fibercast is valued not only for high heat and corrosion resistance, but also for its remarkable maintenance of the purity of the solutions it is required to carry. Such installations range from Fibercast pipes which carry 37% hydrochloric acid at ambient temperatures to lines carrying solutions containing brine, alum, and other sulphates, sulphuric and phosphoric acids, and other damaging chemicals.


1. Smooth interior with a Hazen-Williams C Flow Factor-147
2. Multiple layer glass reinforcement
3. Epoxy coating seal on all cut ends
4. Molded threads
5. Entry lead for easy make-up
6. Coupling stronger than the pipe
7. Standard O.D. pipe size
8. Wrench ring (optional)

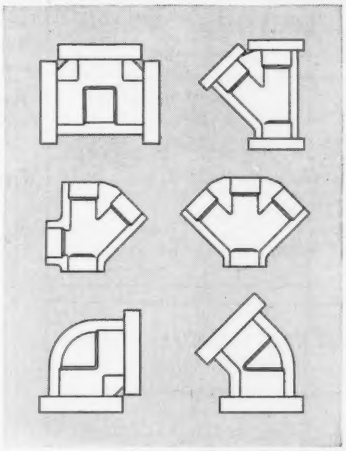
8 Points of Fibercast Superiority

Food-processing industries, among the most demanding of all industries in purity, corrosion and heat standards of operation, know the long-term benefits of using Fibercast. Fibercast is especially effective in the processing of foods requiring use of sulphuric and phosphoric acids (such as corn syrup, starches, gelatins), as well as brines and other highly corrosive acids and solutions.

The operating conditions peculiar to the man-made textile and paper processing plants are also improved by the use of Fibercast Pipe and Fittings. The ever-growing demands of all industry . . . the rigid quality controls imposed by governmental and other agencies . . . the unique and often unsolved needs of new processes and methods . . . all of these factors help to create "impossible" problems that can be solved by the use of Fibercast.

Complete Line of Fittings

Fibercast provides fittings to solve any fitting problem. Besides a vast stock of standard sizes and types, Fibercast also designs and makes special fittings to meet individual requirements. All, of course, have the same corrosion, pressure and heat resistance properties of Fibercast Pipe and Tubing. A few of the regularly stocked types are shown in the line drawings below



If *you* have an "impossible" corrosion, temperature or pressure problem, consider Fibercast. This remarkable pipe can save you money as it has so many other industries. Get the full story without obligation now. Simply mail this coupon.

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Aluminum and Zinc Coatings Compared

(Continued From Page 15)

outside the pole; the galvanized screw was still in good condition. Two more years of exposure completely removed the zinc from the steel strap while the aluminum coated specimen appeared approximately the same.

Figure 2 shows a similar aluminum coated strap exposed on the Bermuda Coast for three years. The severe discoloration is a surface condition attributed to a small amount of iron impurity in the aluminum coating. No pitting was observed. After four years' exposure, the specimen appeared cleaner than shown in the photograph. This history was common to all aluminum coated specimens tested. The corrosion data have been developed from isolated single pieces. However, if aluminum coated material were tested in contact with copper or brass as service requirements sometimes dictate, galvanic action would change the results substantially.

There are, however, serious limitations to the process as presently practiced. Long, unsupported components of relatively thin section are liable to severe distortion when subjected to the high temperature of the molten aluminum bath. This temperature (1320 F) is so close to the lower critical temperature of the steels used that slight variation of the bath temperature can cause erratic changes in the steel's microstructure. As an example, lock washers of high carbon steel, hot dip aluminum coated, sometimes are embrittled to the extent that they snap when flattened or at other times are softened, indicating a tempering effect from the aluminum bath. This same condition has been observed in high strength bolts and insulator polepins. Other problems associated with hot dip aluminum coatings include the scarcity of job shops available, the high installation cost of the process and the high cost of the coating itself.

The per-piece price for hot dip aluminum coated parts is about twice that of galvanized steel. Thus, the aluminum coating fails to satisfy the evaluating criteria from the standpoint of cost, universal corrosion resistance, adaptability and compatibility.

Zinc Filled Coatings

Zinc filled coatings consist of very high percentages of zinc pigment in various vehicles that can be applied by brush, spray or dip methods. These coatings are highly resistant to corrosion. In accelerated tests, some have shown longer life than galvanized coatings. Test panels of one zinc coating exposed on the Bermuda Coast have shown less attack than duplicate galvanized test panels after three years' exposure. Figures 3 and 4 show examples of these panels. This same coating exhibited good compatibility with all common hardware materials except copper and copper alloys.

Figure 5 is a fuse cutout assembly in which the steel components were coated with a zinc-rich pigment coating and then exposed at the Bermuda test area. Arrows in the figure indicate the deterioration of the zinc pigment coating in areas close to the copper contact springs at the top and in contact with the bronze hinge part at the bottom. Salt spray tests in which coated panels were exposed in contact with copper and copper base alloy panels showed the zinc pigmented coating permitted more rusting than galvanized panels exposed in the same manner.

Manufacturing problems involved with zinc coating systems are (1) parts must be handled individually, thereby excluding, from an economical standpoint, their use for large quantity runs of small parts and (2) threaded sections such as on bolts and nuts become clogged, necessitating cleaning.

Because of the lack of compatibility, higher costs and limited applicability, zinc coatings cannot be considered for universal application on hardware products.

Sprayed Metal Coatings

Sprayed aluminum or zinc coatings have been used for corrosion protection successfully in other applications. Relative to hardware products, however, the cost is very high and manufacturing problems are similar to those of the zinc pigmented coatings.

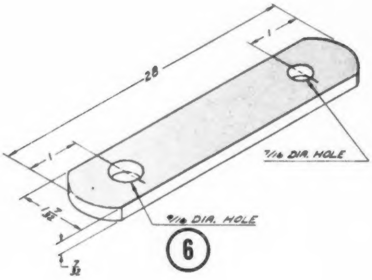

Figure 6—Flat cross-arm brace designed to compare manufacturing costs. Material was mild steel; finish was hot dip galvanized.

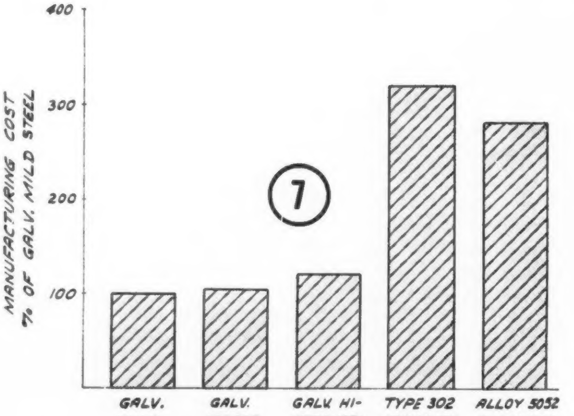

Figure 7—Comparative manufacturing costs of flat cross-arm brace.

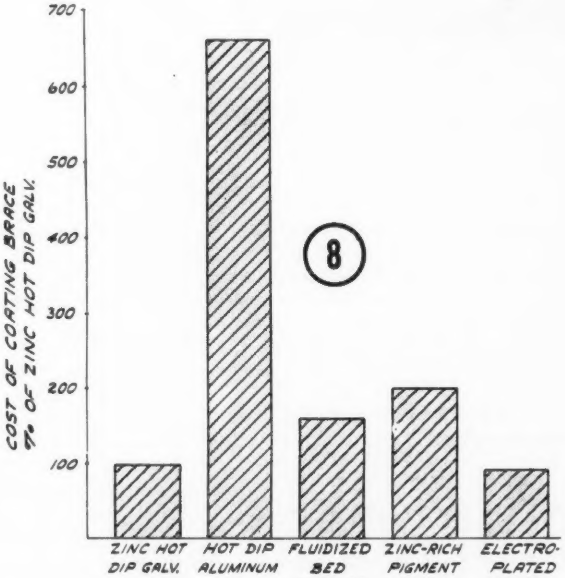

Figure 8—Costs of coating a flat cross-arm brace.

Fluidized Bed Process

The fluidized bed method of applying protective coatings involves suspending resin particles in a container with extremely fine air jets beneath the resin to put them in suspension so they act essentially like a fluid. Metal parts are coated by first heating them to a specified temperature and then immersing them in the fluidized resin.

Coatings applied by this method have had good acceptance where economically applicable. Manufacturing problems associated with the process are similar to those of the zinc pigmented coatings. The cost is approximately twice that of galvanizing.

A further disadvantage of this process, as far as hardware application is concerned, is the lack of sacrificial protection by these coatings. Thus any breaks in the coating result in corrosion of the base metal. The properties of the coatings are, of course, dependent on the particular resin used.

Cost Comparison

For further evaluation of the materials and coatings under discussion, a comparison was made of the manufacturing cost of a simple crossarm brace as pictured in Figure 6, made of various materials and coatings.

Figure 7 illustrates variations due to materials and indicates the relatively high cost of stainless steel and aluminum in an extremely simple application. For other products, where forming operations might be involved, the cost disadvantage for the stainless would be considerably greater. Relative cost of the high strength-low alloy steel also would be increased.

Figure 8 shows cost comparison of various coatings on this type of brace. The high cost of the aluminum hot dip process is apparent here.

Conclusion

So far it is evident that none of the materials or protective coatings considered for longer hardware life are fully satisfactory. Good results can be achieved for isolated applications, but a complete line of corrosion resistant products is still unavailable.

Based on the evaluation presented, the industry must still look to hot dipped galvanized mild steel for today's construction. Recognizing that galvanized steel remains as the most practical hardware material, the producers are continuing research programs to improve the galvanizing process. This work continues while an intensive search is made for a replacement system.

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Failure of Chrom-Moly Stainless Steel Line in a Thermal Cracking Unit*

H. M. Wilten
Lamar State College of Technology
Port Arthur, Texas

Figure 2—Large grain structure with precipitated carbides along grain boundaries found in failed 1½ inch pipe. Etch glyceregia 675X.

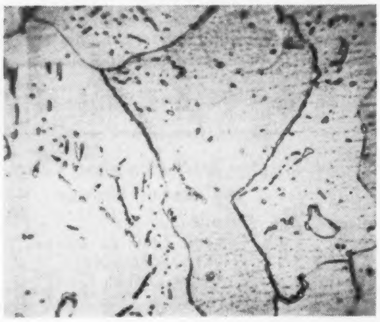
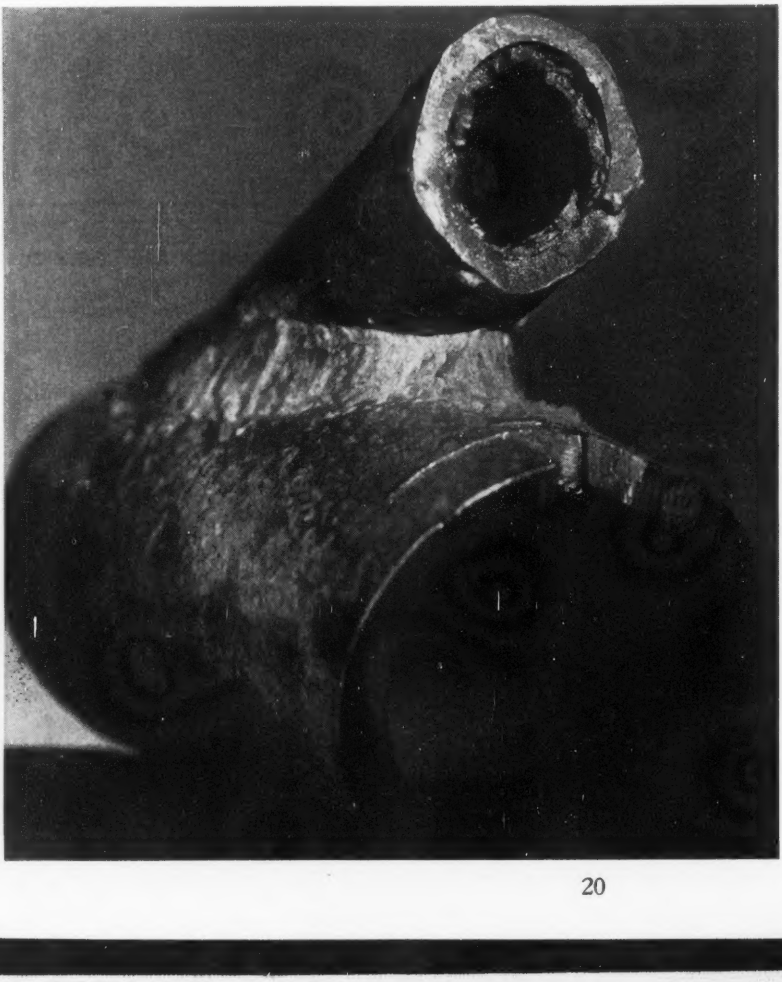


Figure 1—Showing fractured 1½ inch chrome-moly pipe welded into 3-inch line.



FIRST CASE of brittle failure of chromium-molybdenum steel in Texaco oil refinery service in more than 30 years of use is reported here. The 1½-inch 9 Cr line, in service about seven years at 850 F and 400 psi pressure, failed suddenly on down time of the unit. The fracture (see Figure 1) was within a few inches of the place where it was welded into a 3-inch line, of about the same analysis.

In seeking the cause of failure a section of the 3-inch line was flattened until inside walls met without cracks inside or outside. Sections from the 1½-inch pipe were brittle, developing cracks on the inside surface after only minor deformation by a steam hammer. When reannealed at both 1700 and 1350 F, sections of both pipes were flattened without cracking.

Microscopic examination (see Figure 2) showed the 1½-inch tubing had segregated carbides along grain boundaries, while that of the 3-inch pipe showed normal structure.

It is to be noted that the 1½ and 3-inch pipes were subjected to the same service environment. Investigation revealed that during the down-time the 1½-inch pipe was heated with a gas torch and severely hammered to dislodge coke; the 3-inch line was cleaned with a pneumatic tool. Large sized grains at the fracture indicated that the pipe was heated to a relatively high temperature, forming austenite which on cooling transformed to martensite. In a highly alloyed steel such as this, the austenite may still be present in considerable amount after cooling. This retained austenite will transform to martensite when reheated in service. Because martensite is less dense than the parent austenite, a strained and embrittled metal was the result.

DISCUSSION

Comments by R. W. Manuel, Socony Mobil Oil Co., New York City, New York:

Grain boundary carbides are not necessarily an explanation in themselves of the brittle failure. Molybdenum should prevent ordinary temper brittleness in the alloy described. Nothing is visible in the micrograph to support the theory of transformation of retained austenite.

Some difficulty with brittleness has been reported in annealed 12 Cr and even 9 Cr, 1 Mo cast furnace fittings, but these involved segregation that should not be found in a wrought alloy. The 9 Cr cast alloys were, furthermore, on the high side in Cr content in some cases.

The failure described by Mr. Wilten may have involved an age embrittlement phenomenon at 850 F after overheating by torch decoking had made it susceptible.

Causes of embrittlement are many and processes involved often still obscure. It is not clear how this failure should be classified.

★Extracted from minutes of a meeting of NACE Technical Unit Committee T-8 on finery Corrosion held during 16th Annual Conference, National Association of Corrosion Engineers, March 14-18, 1960, Dallas, Texas.

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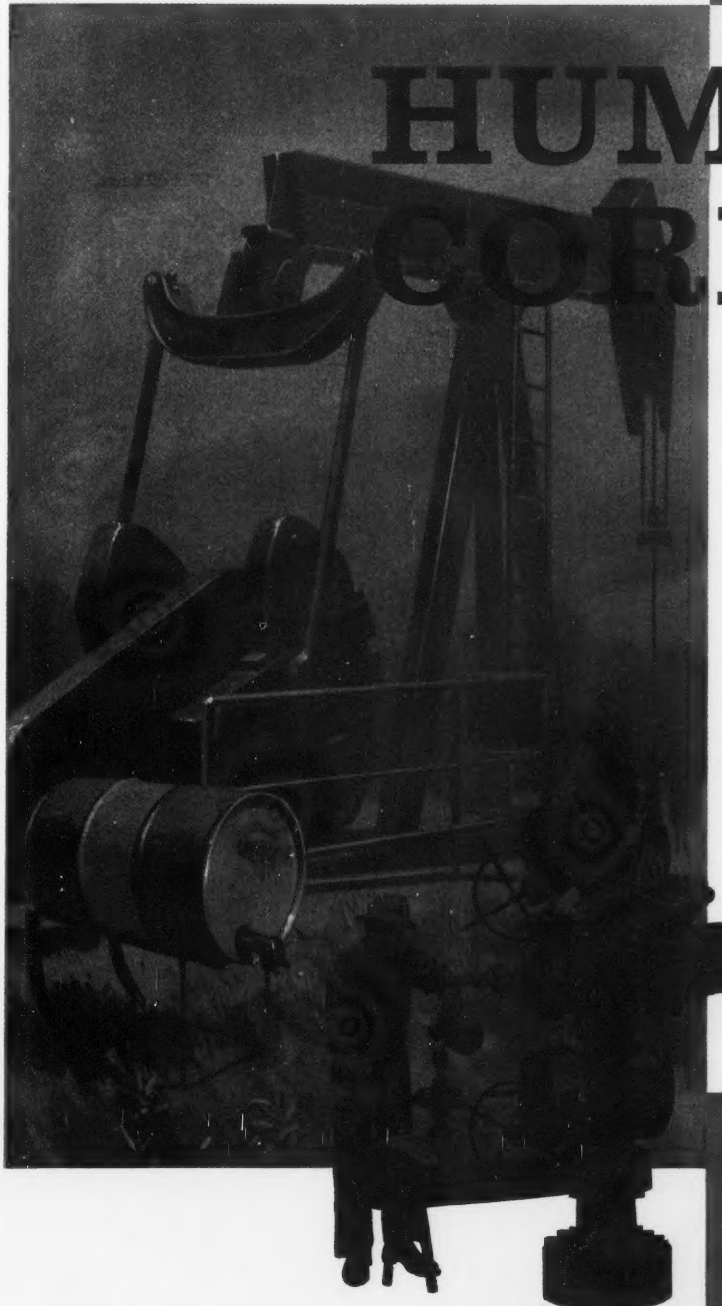

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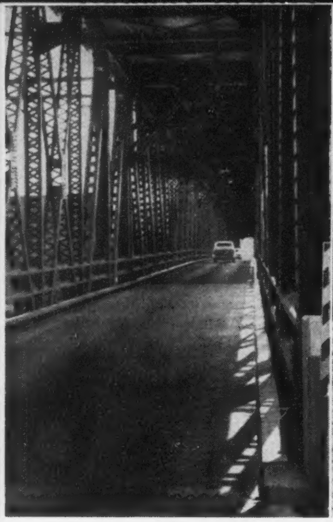

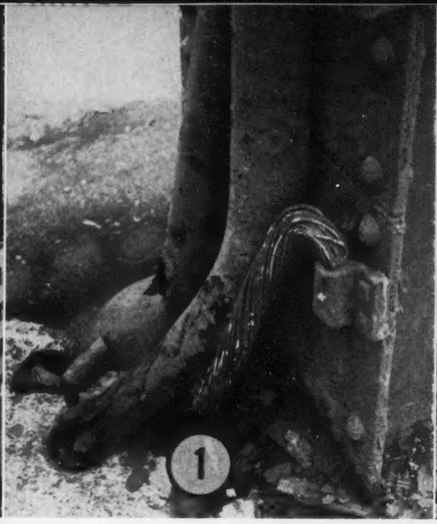

Figure 1—Footing for an elevated railroad in Chicago, showing corrosion attack on steel, the grounding cable and sheathing at base of the structure.

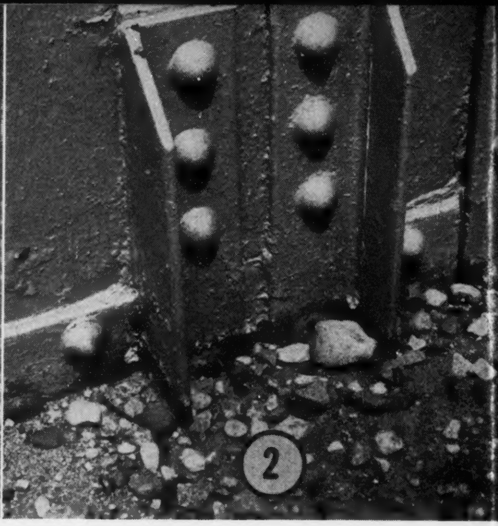

Figure 2—Corrosion attack and paint blisters on footing of highway bridge in South Dakota across the Missouri River.

Figure
River a
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Highway Corrosion P

HIGHWAY CORROSION problems are caused by splashing of water, snow, de-icing salt and mud on steel structures adjacent to the roadway. Resulting accumulation of debris is supplemented by dust collected on the steel structures during dry weather. This affects the lower horizontal flanges of girders, bottom chords and lower portions of steel structures adjacent to the roadway. These accumulations also cause corrosion attack on floor members, shoes, bearings and under-parts of bridges and overpasses, as shown in Figure 1, 2 and 3. Roadway guard rails also are subject to severe corrosion attack plus abrasion from sand and gravel. Figures 4 and 8 show other corrosion problems on bridges.

Other metal structures involved in highway corrosion include hand railings on bridges, cross walks and overpasses, heavy guard rails, road signs and other miscellaneous structures. (See Figures 5, 6 and 7.)

Paint systems for these various structures must be designed for optimum pro-

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★ Extracted from a paper titled "Corrosion Problems in Highway Maintenance" by Nathan W. Morgan, Bureau of Public Roads, Washington, D.C., presented at a meeting of the Baltimore-Washington Section, National Association of Corrosion Engineers, February 16, 1960.

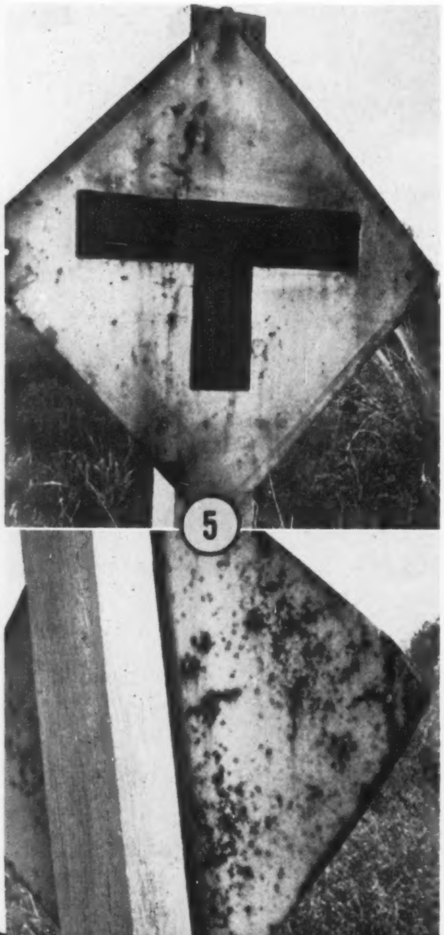


Figure 5—Corrosion and vandalism have almost destroyed this highway sign. Several pistol or rifle shots can be seen on the road intersection symbol, causing paint film to spall on both sides.

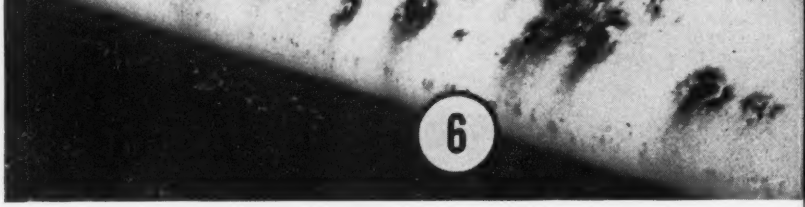
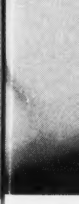


Figure 6—Severe corrosion occurred on this guard rail on a turnpipe highway in Kansas. The corroded areas showed no sign of abrasion or impact to cause the film failure.



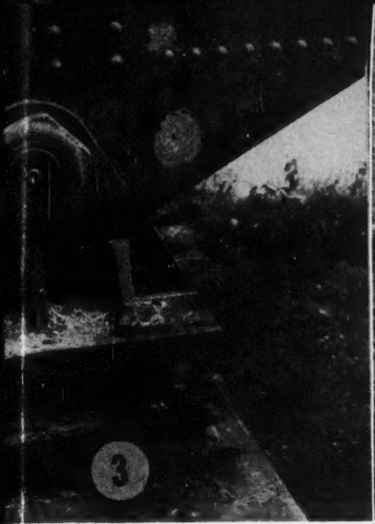


Figure 3—Bearing and footing of highway bridge across the Missouri River on Nebraska-South Dakota border is an example of paint failure and accumulation of dirt and other contaminants on bridge footing.

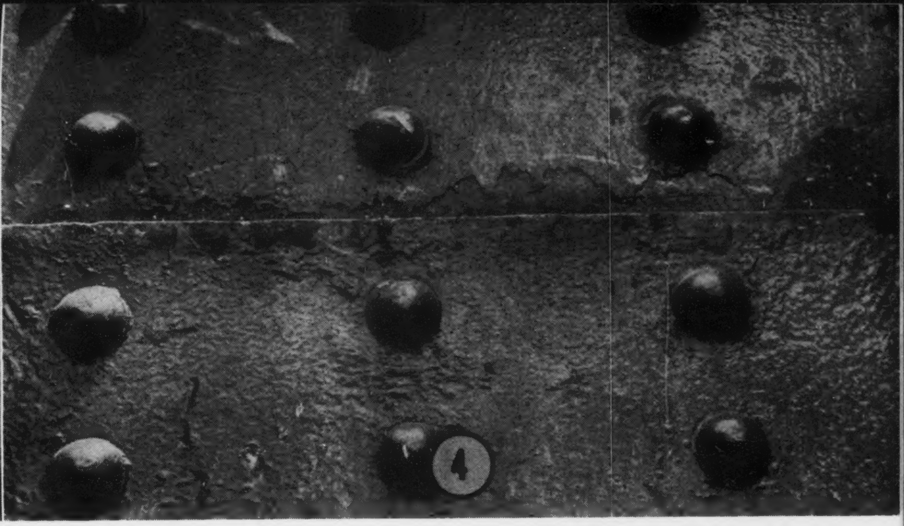


Figure 4—Corrosion caused paint failure on this vertical bridge member. Dirt accumulated on the horizontal ledge formed by overlapped steel plates. Close examination revealed that the surface had not been properly cleaned before re-painting.

Problems*

Caused by Variety of Attacks

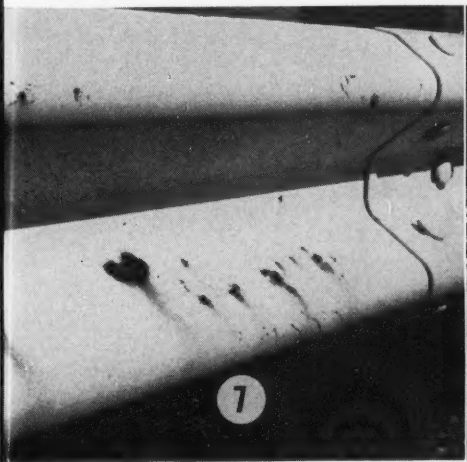
tection for service conditions expected. These conditions may vary from dry to damp rural locations and from heavily polluted industrial areas to the salty high humidity of coastal environments. Corrosive conditions also can include exhaust blast from automobiles, trucks and locomotives, abrasions, water, de-icing salts and even underground corrosive elements such as industrial waste water and electrolysis from cathodic protection systems.

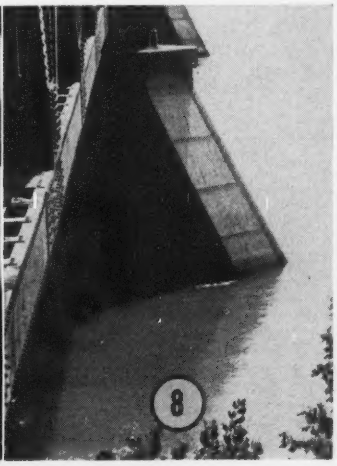
Some industrial atmospheres reduce paint life on highway structures because of sulfur, hydrogen and other compounds which collect in the moisture and dirt on steel structures, resulting in penetration of the paint film.

De-icing salts in solutions ranging to about 5 percent can be the cause of an aggravated corrosion attack. Solution to this problem probably lies in careful design to minimize crevices and cracks where salt and water can be collected. Also corrosion resistant steel and top coats of paint of high impermeability with synthetic resin instead of the ordinary linseed oil vehicle should be considered.

Underground corrosion is a highway problem on culverts and pipes. Soil acidity and stray electric currents complicate this problem. Engineers should be certain that adequate drainage is provided for steel placed underground and insulation should be provided where stray electric currents are encountered.

Public highway property must be maintained or the tax load to replace expensive highway structures will increase.





HIGHWAY
CORROSION
PROBLEMS

Figure 7—Impact from flying gravel and stones may have caused the two large corroded areas where the paint had been knocked loose.

Figure 8—Steel plates mounted on the V-shaped facing of concrete supports on this Missouri River highway bridge at Yankton, South Dakota, present a corrosion problem from alternate wet and dry cycles as the river rises and falls and from abrasion of debris in the water.

Corrosion Tests on Prestressed Concrete Wire*

Howard J. Godfrey
John A. Roebling & Sons Corporation
Trenton, New Jersey

Abstract

Discusses tests conducted to show superiority of hard drawn, stress relieved steel strand over oil tempered strand for prestressing element in concrete structures. Also describes 3-year test showing corrosion caused on steel strand when calcium chlorides were used in concrete mixes for prestressed beams. 2.3.7, 6.6.5

HIGHWAY
CORROSION
PROBLEMS

FEW CORROSION problems have occurred in prestressed concrete construction in the United States since the first large structure was built about 10 years ago. But experience and laboratory investigations have shown that under certain conditions, corrosion can occur and cause severe damage to the steel tensioning elements.

This article illustrates some of the conditions which should be avoided to prevent corrosion of the steel tensioning elements before and after the steel is placed in the concrete.

Investigations of corrosion on prestressed concrete steels have shown that, when a highly stressed steel wire is subject to certain types of corrosion, failure can take place in a distinct manner as illustrated in Figure 1. This type failure is stress corrosion cracking and is characterized by a distinct crack rather than a gradual corrosion of the wire surface.

Oil tempered wire fails much more readily under stress corrosion conditions than does hard drawn stress re-

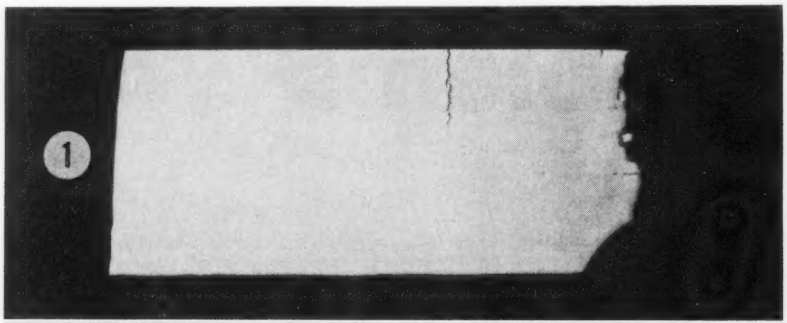


Figure 1—Stress corrosion failure of steel wire used in prestressed structure. (Longitudinal section).

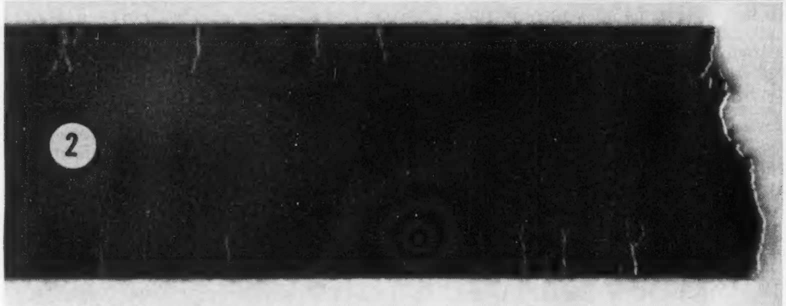


Figure 2—Longitudinal section through failure in oil tempered wire after 38 days in nitrate solution and under 117,000 psi tensile stress.

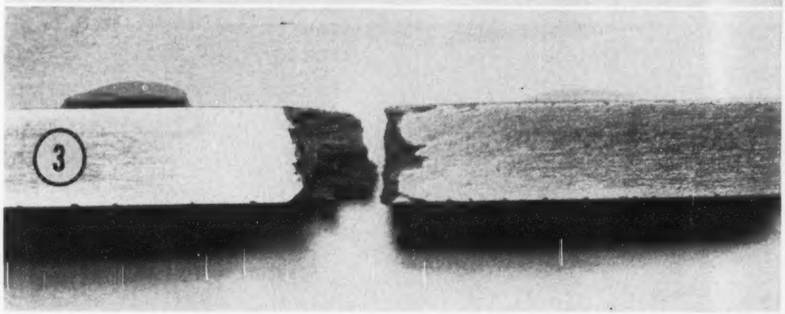


Figure 3—Failure of hard drawn, stress relieved wire (longitudinal section) after 650 days in nitrate solution and under 151,000 psi tensile stress.

★Extracted from *Journal of the Prestressed Concrete Institute*, **5**, 45-51, (1960) March.

lieved wire. For this reason, oil tempered wire is not recommended for use in prestressed concrete.

Laboratory Tests Conducted

Laboratory tests were conducted to prove the superiority of hard drawn, stress relieved wire over oil tempered wire.

Test specimens were stressed in direct tension to 60 percent of their catalog strength and at the same time placed in a nitrate solution containing 600 grams per liter of calcium nitrate and 40 grams per liter of ammonium nitrate. A constant stress was maintained through the test with the corrosive solution at room temperature.

Results of this test are shown in Table 1. Figure 2 shows a longitudinal section through the oil tempered wire at the fracture point. Many transverse cracks developed near the fracture point during the 38-day test.

Figure 3 shows a section of the hard drawn, stress relieved wire which failed after 650 days. This wire was badly pitted but did not develop any deep penetrating cracks. The resting and pitting on this wire were similar to corrosion that occurs after long exposure to weather conditions.

These tests emphasize the superiority of hard drawn, stress relieved wire and also show that both types of wire should be protected from severe corrosion conditions. It is generally assumed that once the steel is covered with concrete that corrosion problems can be forgotten. This investigation, however, indicated that certain precautions should be taken in the use of admixtures in the concrete mix to prevent corrosion.

Precautions With Concrete Mixes

Calcium chloride is used as an additive in concrete mixes to increase the hardening rate. Results of a study on calcium chloride's effect on the corrosion of steel wire and strand are described below.

The concrete mix used contained 7.5 bags of cement per cubic yard of concrete and 5 gallons of water per bag of cement. One series of beams was made without calcium chloride and a second series of beams with 2 percent calcium chloride (2 percent of cement content). Strength of the concrete as determined from 2-inch cubes at the age of 6 months was 6000 psi.

Beams used for the tests were 3 by 3 by 48 inches and contained one $\frac{3}{8}$-inch, 7-wire prestressed concrete strand placed in the center of the cross section. The steel strand was pretensioned to 14,000 pounds or 175,000 psi before the concrete was poured.

The beams were exposed in an industrial atmosphere on the laboratory roof for three years. Then one set of each type beam was broken for examination of the steel strand. As shown in Figure 4, the strands from the beam containing no calcium chloride in the concrete mix had little or no indication of corrosion. Strands in the beams with concrete containing calcium chloride were corroded and severely pitted, also shown in Figure 4. Strands were corroded along the entire length of the 4-foot beams.

Most of the corrosion occurred on the underside of the strands. Corrosion was observed on the center wire of the strands as well as on the six outside wires. Strands taken from both sets of beams were tested for tensile strength. Results are given in Table 2.

Tensile tests on strand from beams without calcium chloride showed no loss in strength or elongation from the original material. Strands from beams containing calcium chloride showed a 5 percent loss in tensile strength and a 60 percent loss in elongation.

Conclusions

These tests indicate that prestressed concrete beams without calcium chloride in the mix were in a satisfactory condition after a 3-year exposure to an industrial atmosphere. Steel strands in the beams with calcium chloride in the mix were badly corroded, indicating that calcium chloride should not be used in prestressed concrete mixes.

TABLE 1—Stress Corrosion Test on Steel Wire*

Type Wire	Failure Time Days	Type Failure
Oil tempered....	13	stress corrosion
Oil tempered....	38	stress corrosion
Hard drawn, stress relieved..	650	rusting, pitting

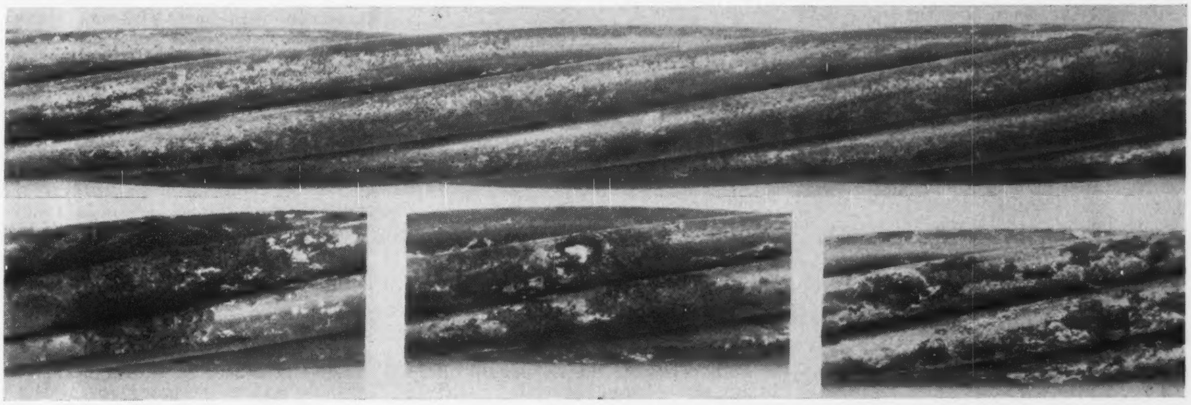
* 0.192-inch diameter.

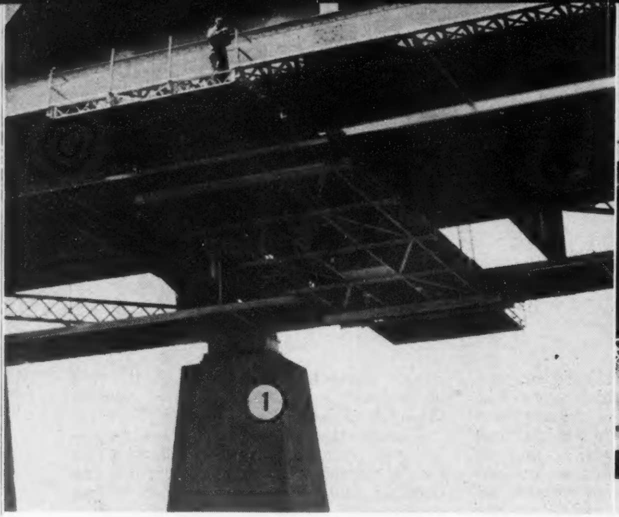
TABLE 2—Tensile Tests on $\frac{3}{8}$-inch Diameter Prestressed Concrete Strands After 3 Years in Concrete Beams

Specimen No.	Concrete Type	Breaking Strength/lb.	Percent Elongation in 24-In.
1-A-1	No calcium chloride	22,550	6.7
2-A-1		22,650	—*
1-B-1	With calcium chloride	21,350	2.6
2-B-1		21,500	—*

* Elongation not determined.

Figure 4—Prestressed concrete strands removed from concrete beams after 3-year exposure in an industrial atmosphere. Top wire was in concrete containing no calcium chloride. Bottom segments were in concrete with 2 percent calcium chloride in the concrete mix.





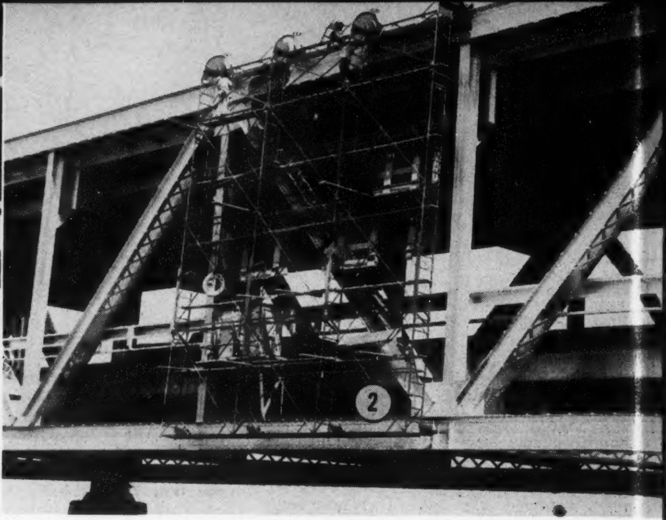


Figure 1—Lower deck travelling scaffold, air powered, gives full access to the bottom deck across full length of the bridge. Figure 2—Truss webb scaffold on the East Bay spans of the San Francisco-Oakland Bridge gives access to deck truss and suspension stiffening truss portions.

Bridge Coating

HIGHWAY
CORROSION
PROBLEMS

Introduction

TOTAL COST for painting the five toll bridges operated by the State of California across San Francisco Bay was about $1,200,000 for the fiscal year 1959-60. An average crew of 115 men applied a total of 29,000 gallons of paint during that year.

These figures indicate the size and importance of corrosion control measures to protect the five gigantic structures linking highways across the San Francisco Bay.

Maintenance and operation of these state owned bridges has been delegated to the Division of Highways by the California Toll Bridge Authority. Names of the bridges are Benicia-Martinez (under construction), Carquinez, Richmond-San Rafael, Dumbarton, San Mateo-Hayward and San Francisco-Oakland Bay Bridges.

These bridges are constructed primarily of structural steel with the exception of the San Mateo-Hayward and Dumbarton Bridges. Because these bridges are subjected to salt water exposure, the major activity of the Toll Bridge Authority has been prevention of corrosion of the structural steel.

Maintenance on these bridges is a year-round project. Much time is lost because of weather conditions which can prohibit any operations by the crews. About 21 percent of the total time is lost due to weather and time spent on activities other than productive painting work, including delays because of bridge traffic and setting up scaffolding and other equipment.

Painting Crews and Equipment Used

Permanent painting crews are assigned to paint the bridges. Two separate crews of about 30 men each paint the San Francisco-Oakland Bay Bridge. One crew covers the West Bay suspension spans; the other crew the East Bay cantilever and deck truss spans. The Richmond-San Rafael Bridge also has two crews of 16 to 20 men. About 10 men handle the painting at the Carquinez and a seven-man crew handle the San Mateo-Hayward and Dumbarton Bridges.

Considerable crew time is spent in rigging scaffolding and other equipment shown in Figures 1 through 4. These special scaffolds have been developed to reduce rigging time and to provide a safe, convenient working platform for the crew. Air powered travelling scaffolds provide full access to the bottom of both decks for the full length of the bridge, as shown in Figure 1.

Truss webb scaffolds shown in Figure 2 permit access to deck truss and suspension stiffening truss portions of the bridge.

Suspender cables in the suspension spans are reached by air-powered scaffolds shown in Figure 3. Scaffolding shown in Figure 4 is all air-powered, giving ready access to all tower surfaces with a minimum of manpower.

The importance of good scaffolding of a permanent nature for an activity of this kind cannot be over-emphasized. Nothing can be done about the time lost to bad weather conditions, but painting efficiency and safety can be improved through development of proper scaffolding designed to fit the structure.

Most scaffolding on the San Francisco Bay bridge is moved by air power. Most repair tools also are air-powered. This air powered equipment plus sandblasting and hand tools require a large, steady and dependable air supply. A new air supply system with a capacity of 3000 cfm will replace the present 2240 cfm system. A new four-inch pipe line extending the length of the bridge will deliver air to the lower deck at strategic points to eliminate most of the portable air supply equipment parked on the traveled way.

Surface Preparation

Surface preparation before paint application on the bridges include steam cleaning, sandblasting, scraping, wire brushing and grinding. Because of its effectiveness and economy, most of the surface preparation is by steam cleaning.

Equipment used for steam cleaning is portable and is designed for washing

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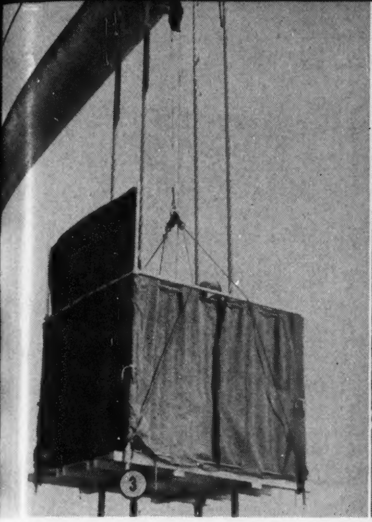
★ Revision of a paper titled "Painting Practices on State-Owned Toll Bridges," presented at a meeting of the Western Region, National Association of Corrosion Engineers, October 6-7, 1960, San Francisco, Cal.



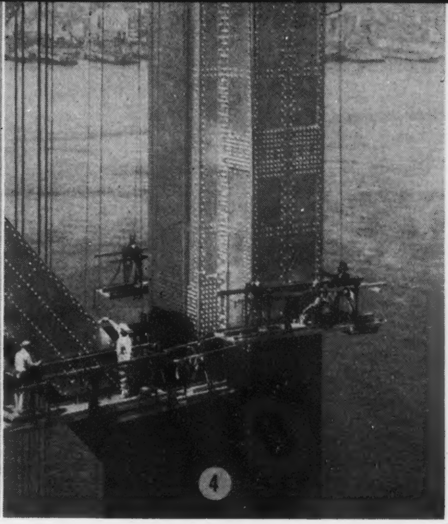


Figure 3—Suspender rope scaffold is air powered, minimizing crew time in raising and lowering scafford as cables are painted. Figure 4—Tower scaffolding also is air powered, giving ready access to all tower surfaces with a minimum of manpower time.

Abstract

Describes paint maintenance program used on state owned toll bridges across San Francisco Bay. Includes cost data on painting operations for five bridges for fiscal year 1959-60. Describes crew, equipment, spot painting procedure, surface preparations and special scaffolding designed to save manpower time.

5.4.5, 1.2.2, 7.10

29,000 Gallons of Paint

Paint Maintenance Program Costs Over a Million Dollars Per Year On Five State-Owned Toll Bridges*

Dale F. Downing and D. Ewing Marsh

Bridge Department, Division of Highways
Los Angeles, California

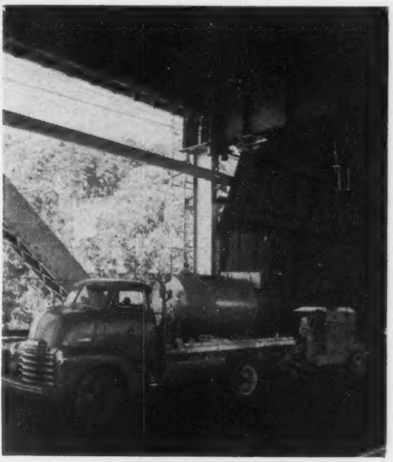


Figure 5—Portable steam cleaning unit. Includes an atomizing burner, feed water supply and feed pump.

surfaces by a heated cleaning solution applied under pressure. The steam cleaner in use is a modified commercial unit consisting of a flash-type heating coil fired by a low pressure atomizing burner with feed-water supplied through a gasoline engine driven feed-pump, all mounted on a suitable trailer. Water supply, to which a cleaning solution has been added, is carried in a tank truck towing the trailer, as shown in Figure 5.

The heated cleaning solution in the form of wet steam is directed on the surface to be cleaned through a hand-operated fan-nozzle.

Figure 6 shows one of the suspension towers being steam cleaned. Figure 7 illustrates the quality of cleaning achieved. Portion of the steel to the right has been steam-cleaned and is ready for painting.

Wet steam is delivered to the nozzle at 125 to 175 psi at 212 F minimum. Under ordinary operating conditions, boiler temperatures will be about 300 to 350 F. Water demand is not more than 3½ gallons per minute. For a normal day's work, an estimated 1200 gallons of water is used per cleaner.

The cleaning compound was developed especially for the San Francisco-Oakland Bay Bridge by the late Carl Hamilton of the Division of Highways and T. K. Cleveland of Philadelphia Quartz Company, Berkeley. The original compound has been modified as follows: (Percentages by Weight)

Sodium Metasilicate (Anhy.)	40
Sodium Sesquisilicate (Anhy.)	35
Tetra Sodium Pyro Phosphate (Anhy.)	10
Alkyl Aryl Sulfonate (40% active)	8
Pine Oil	2
Tetrasodium Ethylene Diamine Tetra-acetate (EDTA)	5

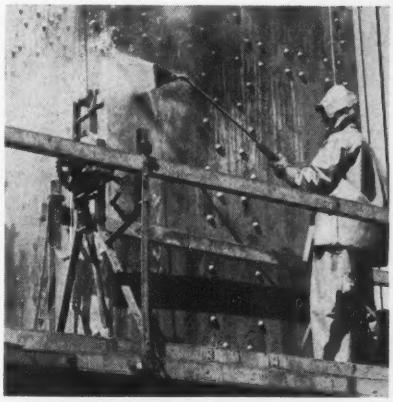


Figure 6—Suspension tower being steam cleaned.

The alkyl sulfonate is a wetting agent. The pine oil prevents dusting and the EDTA is a boiler scale softener. The cleaning compound in solution is added to the feedwater to produce a concentration of ¼ to ½ percent by weight. Stronger concentration merely causes excessive sudsing.

Steam-cleaning removes surface contaminates such as dirt, traffic grime and dead paint which could not be economically removed by other means. Experience has shown that if steam cleaning

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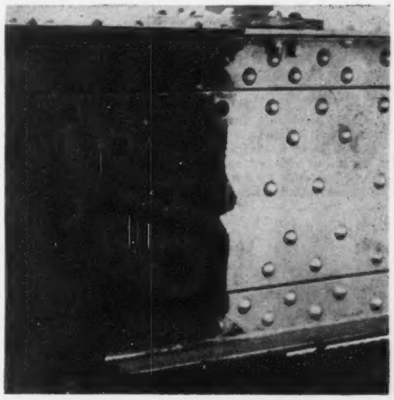

Figure 7—Effectiveness of steam cleaning is shown by portion of beam at right which has been steam cleaned and is ready for painting.

HIGHWAY
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Bridge Coating—

(Continued From Page 27)

is used first to remove dirt and rust stains which appear to be rust, sandblasting can be reduced by as much as 50 percent. Steam cleaning also removes surface contaminates that would impair paint coating performance.

In 23 years' experience, no adverse effects have been found on paint applied over properly steam-cleaned surfaces. Cost-wise, steam-cleaning is about 5 percent of the cost of sandblasting.

Naturally, steam-cleaning is no panacea. Rust, scale and similar tightly adhering contaminates must be removed by sandblasting or other abrasive means. Sandblasting is the most common and widely used on the state-owned toll bridges. In locations where sandblasting can cause problems, power wire-brushing or grinding as shown in Figure 8 is used. Sandblasting near fixed mechanical equipment, over land installations and close to passing traffic is avoided as much as possible.

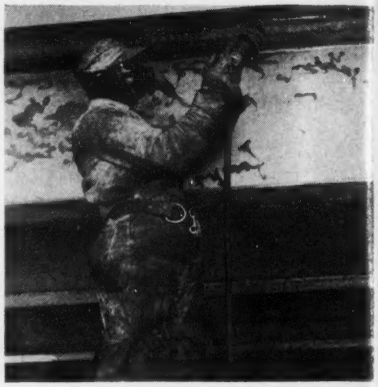

Figure 8—Powered grinders are used instead of sandblasting to remove tightly adhering contaminants where sandblasting can cause problems.

A typical sandblast cleaning is illustrated on the right in Figure 9. The general surface preparation goal is to clean surfaces as thoroughly as conditions permit. It is uneconomical to paint over any surface contaminates, and experience has proved the practicality of sandblasting to gray metal.

Cost and nuisances accompaning sandblasting encourages constant effort to develop other methods. One successful solution is to undertake maintenance painting before deterioration has progressed to the point where sandblasting is required. By practicing this procedure, the Division of Highways has cut its sandblasting requirements in half over the last few years.

Spot Paint Application Methods Used

With the wide variety of exposure and consequent variable service life of the paint coating, complete repainting of the bridges is seldom done. Only areas where paint failure has occurred are cleaned to bare metal. Efforts are made to extend service life of the existing coating, repairing only local weaknesses rather than permitting the failure to progress so that complete replacement of the paint coating would be necessary. This "spot" painting procedure has yielded many dividends in reducing the adverse effects of corrosion.

First step in the spot painting program is to steam-clean all surfaces to remove loose contaminates and dead surface paint. This is followed by spot sandblasting areas where a definite paint failure has developed.

Ordinarily, paint failure areas are sandblasted to bare metal and the cleaned spots are given at least two undercoats of paint. But sandblasted areas now are given a pretreatment of vinyl wash primer before painting to passivate the raw metal. This procedure was adopted from tests conducted jointly by the Materials and Research Laboratory and the Bridge Department in their paint testing program, which disclosed that pretreatment produced an increase in service life by as much as 20 percent on coastal bridges. The vinyl wash primer consists of one part phosphoric acid and alcohol and four parts vinyl buturate. It con-

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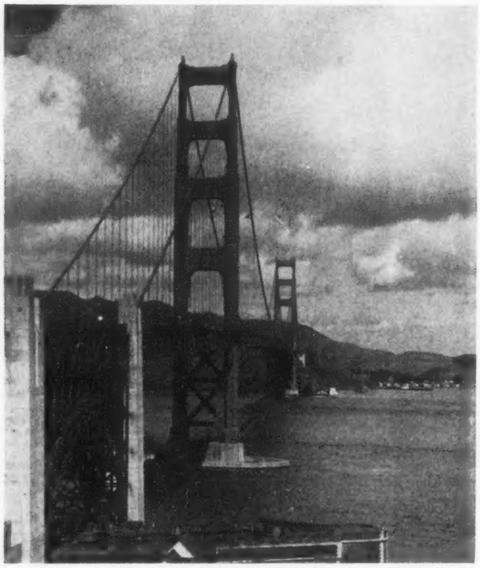
what is "TOOTH"

in a

PRIMER SYSTEM

?



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Bridge Coating—

(Continued From Page 28)

orms to Federal Specification MIL-C-5328.A.

Following spot painting, the steam-leaned area is given a third undercoat o build paint thickness and to insure eplacement of existing paint thickness ost by weathering and steam cleaning. This full third coat improved the apearance of the subsequent finish coat. At locations where the paint failure is airly general, the entire area is sandblasted to bare metal. Here again, a our-coat system is used, three undercoats and one finish coat.

Paint is applied by several methods, depending on the location. Spraying is never used where paint from the spray operation could drift onto passing traffic or fixed land installations. In these areas, paint is applied by brush or roller. Though the paint was not designed for roller application, the finished coating looks good and has more uniform thickness than usually obtained with brushes.

Bottle brushes, sheepskin daubers and paddles faced with carpeting, are standard equipment with the paint crews. There are a great many places on the older structures where these tools are necessary when applying paint to areas that cannot be reached with spray or brush. Some locations require bottle brushes with handles six feet long.

Paint Systems Applied

Basic paint used on the San Francisco-Oakland Bay Bridge is similar to the original type specified during construction. The original paint was a 28-pound linseed oil red lead primer, 27-pound linseed oil tinted red lead second coat and a long-oil phenolic varnish aluminum finish coat.

Basic primer used now is a 27.8-pound linseed oil red lead. The second coat is 24.4-pound tinted linseed oil red lead. Finish coat is the same long-oil phenolic varnish with two pounds of leafing aluminum per gallon of varnish. When two finish coats are applied, the first contains two pounds of non-leafing aluminum per gallon to provide contrast and tooth for the final finish coat.

Several modifications of the basic undercoats are now common practice. Leafed metallic lead often is added to the primer to speed drying and improve the tooth. About 1½ pounds per gallon are added for ordinary use. Because the slow drying characteristics of linseed oil primer often are objectionable, semi-quick dry primer is being used (22-pound red lead in a linseed oil modified alkyd vehicle). This paint dries for recoating in 18 hours, a distinct advantage in many areas. This also reduces possibility of intercoat contamination. Succeeding coats of semi-quick dry undercoats are tinted with lampblack to provide a contrast.

The semi-quick dry paint is used when cleaning is done by sandblasting and fast drying is important. The linseed oil type is used when quality of cleaning must be sacrificed. Frequently, the two are mixed to gain the advantages of each. The other bridges in the Bay Area use semi-quick dry red lead predominately. The same finish coat is used on all bridges now in operation.

Occasionally, special paint systems are applied to meet some peculiar problem. Such a condition exists on the Richmond-San Rafael and the old Carquinez bridges, as described below.

The first few steel girder spans near the west end of the Richmond-San Rafael Bridge are only a few feet above the water. Naturally, corrosion is rapid and severe. Last year the old red lead paint system was completely removed

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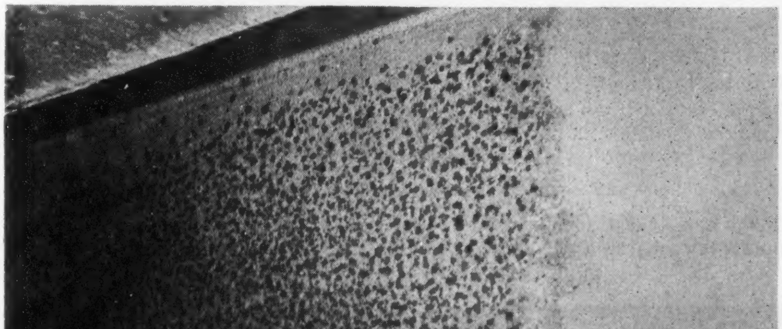
Figure 9—Sandblasted area at right is ready for re-painting. Experience has proved the economic advantages of sandblasting to gray metal before re-coating bridge parts.



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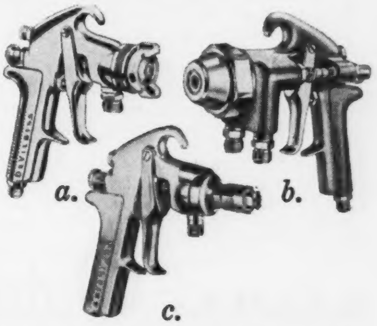

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HIGHWAY CORROSION PROBLEMS

Coating Life Limited Along Riveted Seams

(Continued From Page 31)

by sandblasting and a full vinyl system applied to a minimum thickness of six mils. This system was selected on the basis of experimental applications and full field tests made by the Bridge Department on State Highway bridges along the California coast. Formulation specifications for vinyl paints are now included in the Division of Highways standard specifications.

On the Carquinez Bridge most of the upper chord truss joints have been particularly troublesome. Paint life on inside surfaces has been quite short. These joints are now completely sandblasted and repainted with a vinyl system. This treatment has been confined to inside surfaces because of the difference in appearance between the regular paint system and the vinyl system. If the performance of the vinyl system measures up to the expected standard, a greater use of the vinyl system will no doubt develop. It is doubtful, however, that this system will completely replace the red lead type, due to the difficulty of cleaning and application around traffic.

Trouble Spots Encountered

On bridges the size and type of those across the San Francisco Bay, there is a wide variation in service life of paint coatings. All the trusses, except the new Carquinez Bridge, are riveted structures. The most common trouble spots on these are cracks and seams along riveted connections. There are literally miles of seams in these structures. Examples are the seams along edges of laminated plates of built-up members, seams along stiffener angles, gussett plates of truss joints, joint along shims and filler plates of the major members of the cantilever trusses.

After many experiments in treating these trouble spots, the following procedure has been developed: Cracks are cleaned by sandblasting or digging out rust with hand tools, or both. Then, diluted phosphoric acid and alcohol is squirted into the crack. Next, penetrating oil is applied to deactivate rust deep

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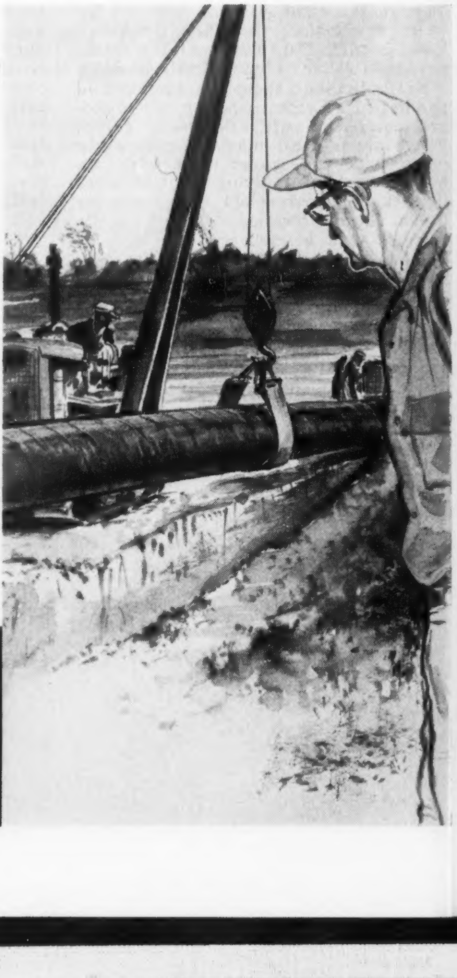
- Caracas, Venezuela



within the crack, which is then sealed with a caulking compound, then painted with normal coatings. Sealing cracks and seams is a constant job. One entire crew spent most of the 1959 season working on cracks and seams on the Richmond-San Rafael Bridge.

The most troublesome general surface area on these bay bridges is the floor system and lower chords of the trusses. The portions facing the prevailing weather show the most rapid failure, which is illustrated in Figure 8.

On the East Bay structure, overhead portions of the cantilever and 504-foot spans are the most troublesome. These areas are painted on the average every four years. Struts, bracing, top laterals and truss diagonals, are built-up members of relatively light sections; consequently, no serious corrosion can be permitted. For this reason, painting operations are almost continual in these areas. Suspension towers, main cables and suspender ropes, give the least trouble. Painting cycle on these members is about 12 years.

One typical area of failure common to all major trusses is the manholes in the chords. For approximately four feet around the manholes, paint on the inside surfaces of box sections breaks down in about two years, attributed to rapid condensation in these areas. The only evident solution is to eliminate the manhole or have additional ones for more rapid and efficient ventilation. On the Bay Bridge, wooden covers have been installed for the manholes in the bottom of the upper chord of the stiffening truss to prevent formation of condensate. The result is that the paint life has been extended about five times.

DISCUSSIONS

Questions by Lee B. Hertzberg, P. O. Box 4616, Oakland 23, California:

1. What was the coverage or cost per square foot?

2. How about using epoxies or polyesters?

3. Was any painting done by contract?

Replies by Dale Downing:

1. Total paint used during the 1959-60 fiscal year on state owned toll bridges was 29,000 gallons. Dividing the total cost figure by this 29,000 gallons gives a cost of $42 per gallon. This does not take into account time spent on other activities which would yield a lesser figure. Over the years, the cost per gallon of paint applied has ranged from $30 to $40 per gallon. Costs per square foot have not been kept due to difficulty of determining the area covered. On contract work (assuming a four-coat job), the costs are comparable. One hundred square feet per gallon at 50 cents per square foot approximately gives a gallonage cost of application of $50 per gallon. These figures must be considered as approximate only. They are a poor comparison and only serve to illustrate that the full time crew can compete with a contract style of bridge maintenance.

2. We have experimented with epoxy and polyester, but so far they do not illustrate a satisfactory advantage. This should not be construed as a rejection. I believe they have fine characteristics for this type of corrosion protection. We have not concluded tests in this field and will continue to investigate. Initial tests were made with early formulations which have subsequently been greatly improved. Results of the early tests show little advantage over conventional systems.

3. No. All painting on state owned toll bridges in California is done by regular full time state employees. Except for a similar crew working on steel structures in metropolitan San Francisco, all other state highway bridges are painted by contract.

Question by James Scheibli, Shell Development Co., Emeryville, California: Were colors other than aluminum used, such as gray?

Reply by Dale Downing:

Pigmented finish coats had two main disadvantages. First they tend to fade with time and type of exposure. This results in a mottled appearance which with some colors becomes objectionable. Secondly, natural accumulations of traffic grime and dirt become very noticeable. If not cleaned, dirty films give the appearance of neglect. The aluminum color was selected because it was neutral. Furthermore, age and exposure merely dulled the finish. Dirt and traffic grime were much less noticeable.

Question by William Sawyer, DeSoto Chemical Corp., Berkeley, California: Were any re-coating problems experienced with vinyl?

Reply by Dale Downing:

We have had very little experience in spot painting vinyl systems. We expect some problems, but early experiments indicate such procedures would be feasible with no great increase in cost.

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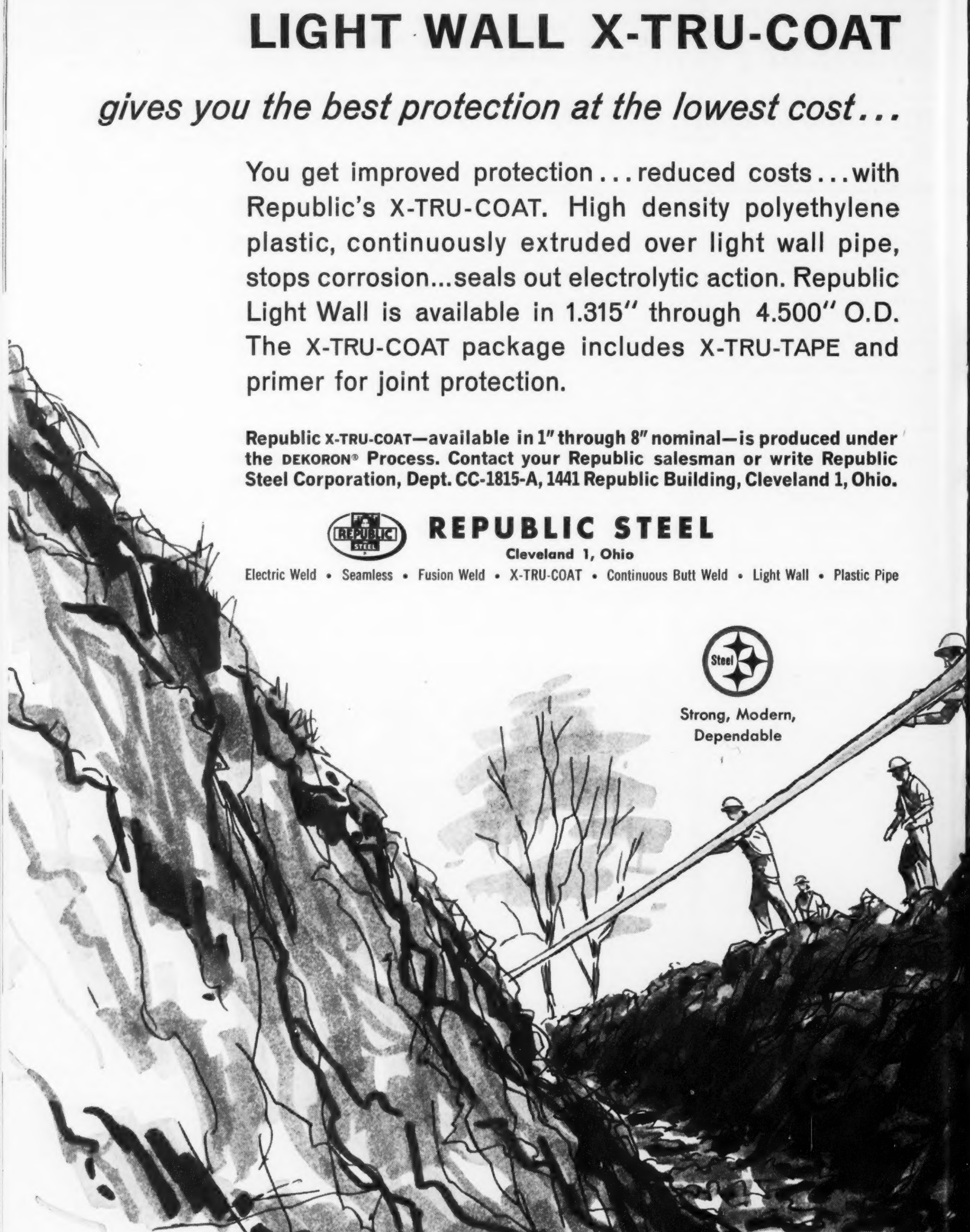

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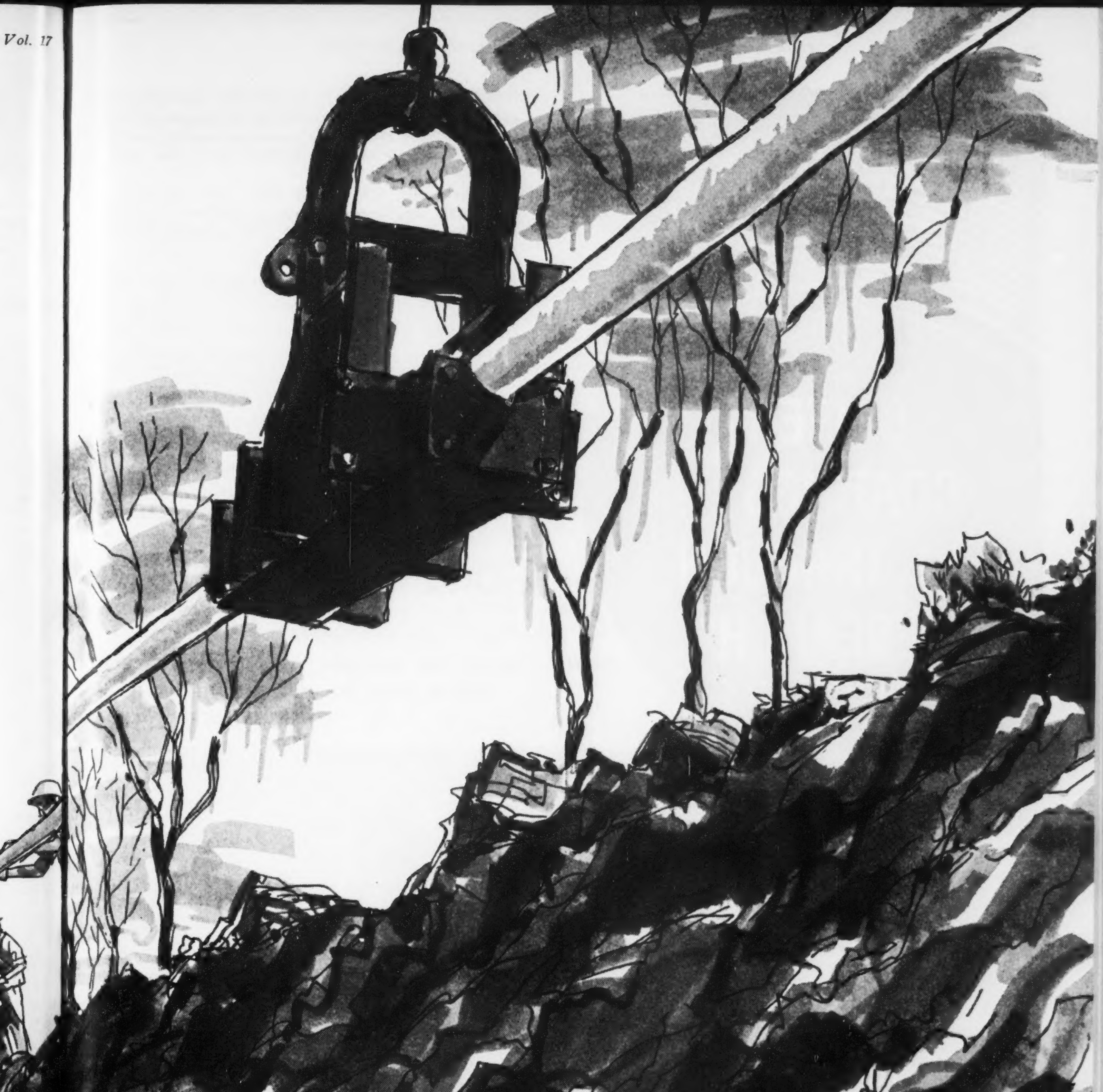

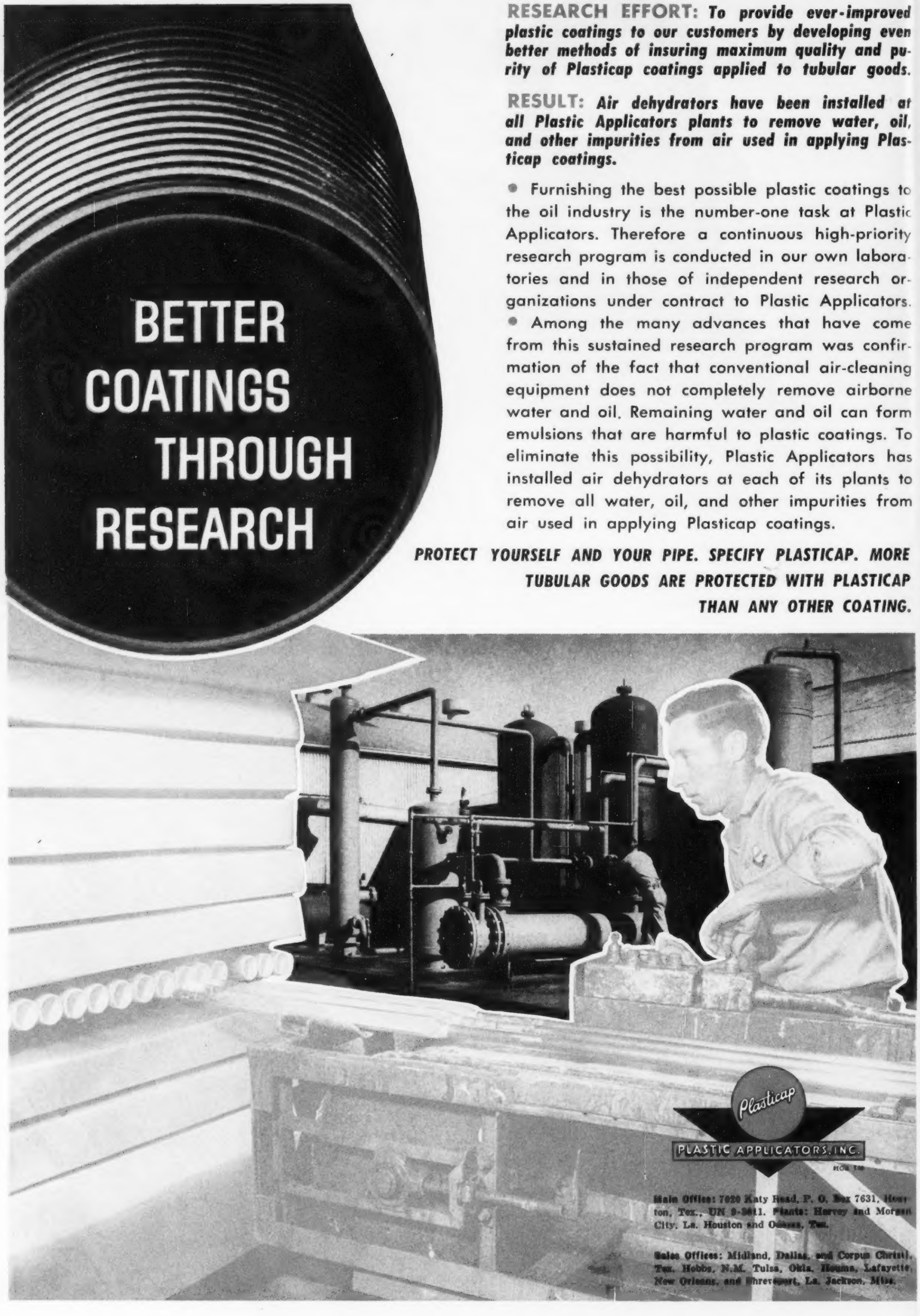

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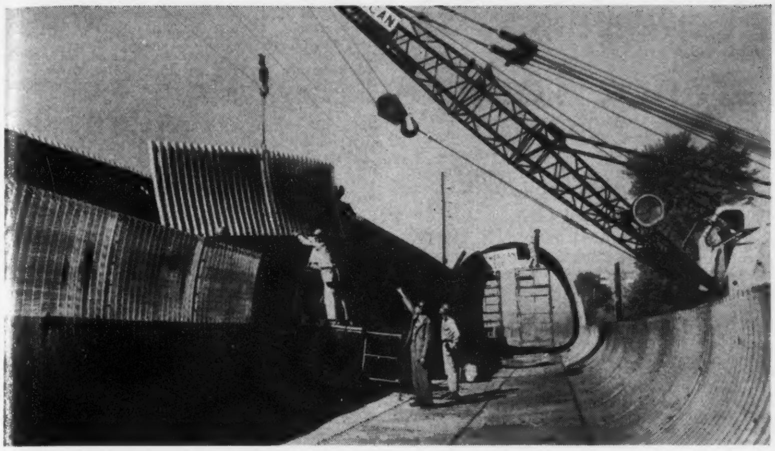
HIGHWAY

CORROSION

PROBLEMS



VEHICLE TUNNEL made of corrugated steel plate sections will have a concrete highway running through it when completed. This will be one of twin tunnels measuring 334 feet long, about 20 feet wide and 17 feet high. Five-gauge copper bearing galvanized steel plates are used. The tunnels are being constructed by U. S. Steel Corporation's American Bridge Division on Interstate Highway 74 near Morton, Illinois.

Estimated 11 Billion Dollars Spent on Highways in 1960

During 1960, an estimated 11 billion dollars was spent on building and maintaining roads and streets in the United States. This amount is more than the federal budget of any peacetime year before World War II.[1]

This 11 billion dollars is only a part of what will be spent on the more than 40,000 miles of new super highways scheduled for completion in the next few years. Some of these highways cost 30 million dollars a mile.[2]

Since 1957, the first full year of the new federal interstate highway system program, the federal government has collected over 14 billion dollars in special taxes on motor fuel, tires, tubes, new automobiles, parts and accessories, lubricating oil, trucks and buses, of which only a little over eight billion dollars was used for highway purposes. Over six billion of this eight billion dollars came from federal taxes on gasoline.[1] This eight billion dollars of tax money is more than half the total value of the products shipped by the petroleum refining industry in 1958.[3]

America's new super highways and freeways have cost the taxpayer an estimated 18.2 billion dollars since the federal highway program was begun in 1956.[1] These highways and the bridges, overpasses, underpasses and other structures including lighting facilities and highway signs must be properly designed and constructed to prevent unnecessarily heavy maintenance which can result from corrosion. Few taxpayers realize that many corrosion problems must be solved to protect highways, bridges, light poles, sign, guard rails and other metallic structures from atmospheric corrosion, damage from precipitation and the accelerated corrosion damage from de-icing salts used on highways in parts of the country.

Corrosion problems encountered in highway structures are just as varied as are the structures: suspension bridges, truss bridges, reinforced concrete structures, prestressed concrete structures, etc. Each has corrosion problems which differ to some extent from the others. For example, a reinforced concrete bridge exposed to a marine atmosphere may suffer serious corrosion damage within a relatively short time because concrete surrounding the reinforcing steel can act as an electrolyte, setting up corrosion cells. Concrete around the steel is spalled off, exposing the steel to more rapid atmospheric corrosion.[4]

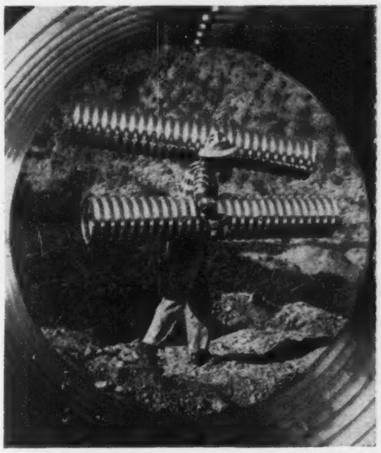
This problem of corrosion of steel in concrete has been discussed in three articles published in Corrosion: (1) Electrolyte Corrosion of Steel in Concrete, by A. E. Archambault, Corrosion, **3,** 37-51 (1947) January. (2) Electrolytic Corrosion of Steel in Concrete, by G. M. Magee, Corrosion, **5,** 378-382 (1945) November. (3) Corrosion of Steel in a Reinforced Concrete Bridge, by R. F. Stratfull, Corrosion, **13,** 173t (1957) March.

Another article on this problem of corrosion on reinforcing steel was published more recently: Corrosion of Reinforcing Steel in Marine Atmospheres, by D. A. Lewis and W. J. Copenhagen. Corrosion, **15,** 382t (1959) July.

Some of the varied corrosion control problems on steel bridges, road signs and other highway structures are discussed in Technical Topics articles on Pages 9-34 of this issue.

Special corrosion problems have been encountered in prestressed concrete bridges. Improper cement mixtures can result in corrosion of the steel wire and cable used to stress structural members.

An idea of the magnitude of the corrosion control problem on the current federal highway program can be obtained by considering the 65,000 overpasses that will be built during the next ten years. Repainting of these overpasses alone is estimated to cost over 20 million dollars a year unless proper design is used and proper materials selected. Some materials are more corrosion resistant than others; some steels will hold a coat of paint longer than others. These and many other factors should be considered if the taxpayer's dollar is to be used more effectively to prevent future costs of repainting or rebuilding highway structures.



LIGHT ENOUGH for a man to carry them is one feature of aluminum culvert pipe which is claimed to have about the same yield strength as mild steel. The culverts are made from corrugated aluminum sheets that are formed and rivited. The culverts have been priced competitively with conventional galvanized steel culvert.

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[1] *Oil Facts,* Vol. 2, No. 5, Oct.-Nov., 1960. Published by American Petroleum Institute.

[2] *Time Magazine,* January 18, 1960.

[3] U. S. Department of Commerce report, May, 1960.

[4] D. A. Lewis and W. J. Copenhagen. Corrosion of Reinforcing Steel in Marine Atmospheres. *Corrosion,* **15,** 382t (1959) July.

RECORD and REPORT

High Temperature Materials to Be April 26-27 Conference Topic

Several papers of probable interest to corrosion engineers are scheduled to be presented at the April 26-27 Technical Conference on High Temperature Materials to be held at the Pick-Carter Hotel, Cleveland, Ohio. The conference is sponsored by the AIME Cleveland Section.

The papers are scheduled as given below.

Wednesday p.m., April 26
Tungsten, Tantalum and Their Alloys

Status and Future of Tantalum, Tungsten and Their Alloys, by H. R. Ogden and D. J. Maykuth, Battelle Memorial Institute.

Mechanical Properties of Arc Melted Tungsten and Some Tungsten Alloys, by F. A. Foyle, Lewis Research, Center, NASA.

Tungsten-Rare Earth Alloys, by C. Thomas, G. Asai and H. Kato, U. S. Bureau of Mines.

Effect of Surface Condition of Ductility of Tungsten, by J. R. Stephens, Lewis Research Center, NASA.

Properties and Fabrication of Tantalum-Base Alloys, by A. L. Field, Jr., R. L. Ammon, A. I. Lewis and L. S. Richardson, Westinghouse Corp.

Physical Properties and Fabrication Techniques for Tantalum-Base Alloys, by M. L. Torti, National Research Corp.

High Temperature Oxidation Behavior of Complex Tantalum Alloys, by W. D. Klopp, D. J. Maykuth and H. R. Ogden, Battelle Memorial Institute.

Wednesday p.m., April 26
Cobalt and Nickel Base Alloys

Status and Future of Cobalt and Nickel Base Alloys, by W. H. Sharp, Pratt & Whitney Aircraft.

Nickel Base Alloys for the 1960's, by C. S. Freer, R. A. Woodall and S. Abkowitz, Kelsey-Hayes Co.

A New Series of High Temperature Nickel-Base Alloys, by J. C. Freche, W. J. Waters and T. J. Riley, Lewis Research Center, NASA.

Effect of Heat Treatment on Cost Age-Hardenable Ni-Cr Alloy, by C. G. Bieber and R. F. Decker, International Nickel Co., Inc.

Effect of Phase Changes on Workability and Mechanical Properties of Udimet 700, by J. Quigg, Thompson Ramo Wooldridge Inc.

Effect of Metallurgical Factors on Creep-Rupture of Alloys at Very High Temperatures, by M. Korchynsky, Union Carbide Metals Co.

Effect of Alloying on Properties of Wrought Cobalt, by J. J. Rausch, J. B. McAndrew and C. R. Simcoe, Armour Research Foundation.

Thursday, a.m., April 27
Molybdenum, Columbium and Their Alloys

Status and Future of Molybdenum, Columbium and Their Alloys, by L. P. Jahnke, General Electric Co.

Mechanical Properties of Cb-N-Mo and Cb-V-W Alloys, by R. T. Begley, Westinghouse Electric Corp.

Properties of Some Columbium-Base Alloys in the Columbium-Tantalum-Tungsten-Zirconium System, by W. O. Gentry and A. B. Michael, Fansteel Metallurgical Corp.

Aging Phenomena in Columbium-Base Alloys, by D. O. Hobson, Oak Ridge National Laboratory.

Development of High-Strength Columbium-Base Alloys for Reactor Cladding Application, by J. A. DeMastry, F. R. Schober and R. F. Dickerson, Battelle Memorial Institute.

Solution and Aging Reactions of Molybdenum-Base Alloys, by W. H. Chang, General Electric Co., and I. Perlmutter, Wright Air Development Division.

Vanadium Alloys for 1800 to 2200 F Service, by B. R. Rajala and R. J. Van Thyne, Armour Research Foundation.

Thursday p.m., April 27
Fabrication of Refractory Metals

Effect of Melting Variables on Purity and Properties of Tungsten, by R. W. Cambell and C. D. Dickinson, General Telephone and Electronics Corp.

Processing and Properties of Columbium-Base Alloy Cb-74, by C. R. McKinsey, A. L. Mincher, W. F. Sheely and M. Schussler, Haynes Stellite Co.

Welding of Columbium and Its Alloys, by E. A. Franco-Ferreira and G. M. Slaughter, Oak Ridge National Laboratory.

Thursday p.m., April 27
Graphite and Refractory Compounds

High Temperature Properties of Titanium Diboride, by V. Mandorf, J. Hartwig and E. J. Seldin, National Carbon Co.

Refractory Beryllium Compounds, by W. W. Beaver and A. J. Stonehouse, Brush Berlyllium Co.

Preliminary Investigation of Fabrication and Properties of Hafnium Carbide, by W. A. Sanders, J. W. Creagh, C. F. Zalabak and J. J. Gangler, Lewis Research Center, NASA.

Thursday p.m., April 27
Coatings on Refractory Metals

Status and Future of High Temperature Coatings for Refractory Metals, by J. J. Gangler, Lewis Research Center, NASA.

Coatings for Molybdenum, by R. L. Wachtel, Chromalloy Corp.

Mechanisms of Formation and Protection of Columbium-Zinc Coatings on Columbium, by G. Sandoz and T. C. Lufton, Naval Research Laboratory.

Protection of Columbium Alloys for High Temperature Service, by E. N. Bamburger, R. G. Frank, L. Luft and E. D. Sayre, General Electric Co.

Ceramic Coatings for Protection of Columbium Alloys, by W. B. Hall and J. W. Graham, General Electric Co.

High Temperature Oxidation Resistant Coatings for Columbium Alloys, by R. A. Jeffries and J. D. Gadd, Thompson Ramo Wooldridge Inc.

Aluminide and Beryllide Protective Coatings for Tantalum, by D. D. Lawthers and L. Sama, Sylvania-Corning Nuclear Corp.

16mm Film Catalog Lists Free Industrial Materials

A catalog of more than 130 films available from Modern Talking Picture Service, Inc., 3 East 54th St., New York 22, N. Y., has been issued and is available on request to businesses, industries, schools and others. The films are offered free to adult groups connected with business or industry, the only charge being round-trip parcel post.

Covering a wide variety of topics from solderless electrical connections, the story of automobiles through the years, recovery of a missile nose cone, modern earth moving equipment, welded steel tubing manufacture, brass making and many others, the 16 mm, sound-on-film motion pictures are sponsored by industrial firms.

Corrosion-Interest Reports Available From Government

Several reports of probable interest to corrosion engineers and persons in the corrosion control field are available from the U.S. Department of Commerce. These Office of Technical Services Reports are listed below.

Physical Properties of Some Nickel-Base Alloys (PB 151086) $2.75

Diffuse Coatings on Iron and Steel (60-21148) $1.75

USSR Literature on Air Pollution and Related Occupational Diseases, Vol. 4 (60-21913) $4.00

New Developments in the Welding of Metals (PB 151089) $1.25

Development of Refractory Metal Sheet in the United States (PB 161217) $.50

Recent Developments in Superalloys (PB 161219) $.50

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Trademarked Geospace, the structures will be produced by Filtered Rosin Products Company, Baxley, Ga., a subsidiary of Monsanto Chemical Company's Organic Chemicals Division.

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Three NACE Members Give Corrosion Papers At TAPPI Meeting

Three NACE members presented technical papers on corrosion at the 16th annual meeting of the Technical Association of the Pulp and Paper Industry (TAPPI) held February 20-23 in New York City.

Wilbur J. Comerford, NACE member with the Alloy Tube Division of Carpenter Steel Co., Union, N. J., gave a paper titled "Manufacture of Welded Stainless Steel Tubing for Kraft Evaporator Service."

A paper titled "Corrosion in Tall Oil Distillation" was given by Harold C. Templeton, chief metallurgist with Alloy Steel Products Co., Linden, N. J.

The third NACE member on the TAPPI program was T. R. B. Watson, president fo Corrosion Service Limited, Toronto, Ontario, Canada. He gave a paper titled "Electrolytic Corrosion Protection of Paper Mill Equipment."

NBS Research Summary

A digested summary of the accomplishments of the National Bureau of Standards is included in "Research Highlights of the National Bureau of Standards", Annual Report 1960. The 189-page book is designated Miscel. Pub. 237 and may be obtained from Superintendent of Documents, U.S. Government Printing Office, Washington 25, D. C. for 65¢.

About 225 programs in 18 different fields are presented with illustrations.

BOOK NEWS

1959-60 NACE Technical Committee Reports. 52 pages, 8¼ x 11¼, plastic binding. Jan. 1961. Publication B-73. National Association of Corrosion Engineers, 1061 M & M Bldg., Houston 2, Texas. Per copy, NACE members, $5; non-members, $7.

Twenty-four reports published in 1959-60 by technical committees of NACE. They include two on corrosion of oil and gas well equipment; one on pipe line corrosion; six classified as general; three on utilities, three on the process industries, four on protective coatings and five on petroleum refining.

All reports previously have been published in CORROSION.

Aluminium. Vol. 37, No. 1, 94 pages, 8½ x 11⅝ inches. (In German.) Aluminium-Verlag GMBH, Dusseldorf, Germany. Subscriptions, DM 3.50

Consists of general information about aluminum and the aluminum industry, with accent on economics. There is a roundup of world information on aluminum in this issue. A section is devoted to commercial notices and another to abstracts.

A substantial part of the serially number part is devoted to technical articles on aluminum.

1960 Supplement to the AWS Bibliographies. Compiled by Edward A. Fenton. 31 pages, 8½ x 11¼, loose leaf. 1960. The American Welding Society, 33 West 39th St., New York 18, N. Y. Per copy $1.50.

A supplement to the original document covering 1937-57 issued in 1958. Sheets may be inserted in appropriate sections of the old index, or kept as issued.

Consists of titles of princpal articles that have appeared in The Welding Journal during 1960 concerning aircraft, aluminum, brazing, destructive examination, inspection and non-destructive examination, magnesium, metallizing, railroad industry, ship construction, stainless steel, and many other categories.

Administrative Report, 1958-59; 1959-60. 20 pages each, approximately. 1960. Administration of Water Works, City of Bombay, India. Availability not indicated.

Extensive and detailed information on the operation of the water works of the city of Bombay for the period 1958-60 is included in the two reports. Maps and other data give an excellent picture of the operation of this facility. Of interest to water works engineers, especially those concerned with systems located in the tropics.

Bibliography on Industrial Radiology 1958-60. By Herbert R. Isenburger. 21 pages, 8½ x 11, typescript, loose leaf. 1961. St. John X-Ray Laboratory, Califon, N. J. Per copy, $4.00.

Consists of references 5912 to 5905 in the continuing series on this subject issued by the publishers. It is the 8th supplement to "Industrial Radiology" published by John Wiley & Sons, N. Y., 1943. Included among the references are those pertaining to the September 1960 Copenhagen Conference on the Use of Radiosotopes in the Physical Sciences and Industry sponsored by the International Atomic Energy Agency.

POSITIONS WANTED and AVAILABLE

Active and Junior NACE members and companies seeking salaried employees may run two consecutive advertisements annually without charge under this heading, not over 35 words set in 8 point type. Advertisements to other specifications will be charged for at $12.50 a column inch.

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Research & Development engineer, registered mechanical. Good chemistry background; 16 yr experience design, testing, application valves; familiar metals evaluation, coatings, plastics, elastomers, adhesives, lubricants petrochemical, chemical process services. CORROSION Box 61-4.

Sales Engineer—Experienced in analyzing corrosion problems, sales of protective coatings & linings, application or installation of materials. Desire sales or service position in corrosion control work. Resume on request. Willing to relocate. CORROSION, Box 61 - 2

Corrosion-Materials-Finishing Engineer — Broad experience-background covering laboratory, technical sales/service in above lines. Heavy supervision and management responsibilities, in manufacturing as well as laboratory. Last 5 years in missile field. Resume on request. CORROSION, 61-6.

CORROSION ENGINEER—E. E. degree. Ten years' experience in corrosion control engineering, design, installation, personnel training, standardization practices for transmission and distribution companies. Experience in sales engineering of corrosion control products. Will relocate. CORROSION, 61-3.

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Metallurgist with corrosion engineering experience wanted for staff position, civil service, project management Army guided missile weapons systems. Write W. H. Ewart, Chief, Engineering Requirements, Coordination Office, Research & Development Operations, Army Rocket and Guided Missile Agency, U. S. Army Ordnance Missile Command, Redstone Arsenal, Alabama.

Chemical Engineer—Knowledge of current water treating practices by top flight, growing water treating company. Man selected will have outgoing personality and administrative ability for future advance. Excellent salary, moving expenses, insurance. Send resume and desired salary. Our employees know of this advertisement. CORROSION, 61-7.

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NACE's 18th Annual Conference and 1962 Corrosion Show will be held at the Municipal Auditorium, Kansas City, Mo., March 19-23, 1962.

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Cleaners

Cleaning scale deposits from boilers, heat exchangers, water lines, pumps and other equipment with a powdered acid cleaner is described in a bulletin available from Hagan Chemicals & Controls, Inc., Hagan Center, Pittsburgh 30, Pa. The cleaner is a free-flowing mixture of a solid acid, corrosion inhibitor and anti-foam, according to Hagan. The active inhibitor is included to make the cleaner safe for use in systems containing iron, steel, copper, copper alloys, brass and aluminum.

•

Importance of chemical cleaning for efficient operation and extended service life of boilers, auxiliaries and heat exchangers is discussed in a 12-page technical booklet available from Dow Industrial Service, 20575 Center Ridge Road, Cleveland 16, Ohio. Another booklet available from Dow describes comprehensive chemical services of the company in missile cleaning, water and waste treatment and processing, laboratory and technical counseling and other services.

•

Newly developed scale dissolution rates for sulfamic acid cleaners are included in a booklet published by Du Pont and available from 4147 Du Pont Bldg., Wilmington 98, Del. Use of sulfamic acid cleaners for in-place descaling of industrial equipment has increased, according to Du Pont, because of their efficiency, ease of handling and safety to personnel and equipment.

Coatings, Organic

Techniques for formulation of vinyl solution coatings are given in a booklet published by Union Carbide Plastics Co., 270 Park Ave., New York 17, N. Y. The booklet gives basic information on general properties, uses, formulations and preparation of clear and pigmented coatings. Application methods also are given. Applications are shown ranging from sulfuric acid processing equipment to vinyl coated book binding.

•

Quick curing resin for producing glossy, durable automobile body enamels without catalyst is claimed by Monsanto Chemical Company's Plastics Division, Springfield, Mass. Called Resimene 872, the new formulation is an etherified melamine formaldehyde resin solution of 50 percent solids content in isobutanol. The material is used with alkyd resins to make baking enamels suitable for finishing automobiles, appliances, equipment housings and other metal products where a wide range of cure schedules are required. Enamels formulated with Resimene 872 can be baked at schedules from 30 minutes at 180 F to 10 minutes at 300 F with minimum gloss differential and high color retention, even when overbaked, according to Monsanto.

•

Marring and scratching of polished, embossed or coated metals during fabrication, storage and shipping can be eliminated by use of a strippable vinyl coating called Strip-Gard, according to the developers, Stoner-Mudge Company of Pittsburgh, Pa., a division of American-Marietta Co., 101 East Ontario, Chicago, Ill. The coating is an organosol based on vinyl manufactured by B. F. Goodrich Chemical Co., 3135 Euclid Ave., Cleveland 15, Ohio.

Fittings

Type 321 stainless steel bellows-type expansion joints to fit standard pipe from 3 to 72 inches nominal diameter are manufactured by Pathway Bellows, Inc., 999 Industrial Place, El Cajon, Cal. The firm's hydro roll folding method permits a wide range of metal thicknesses and convolution depths for high or low spring rates and pressures, according to the manufacturer.

•

No-air inclusion couplings designed for minimum leakage on disconnect and minimum entrapment of air or foreign matter on connect have been developed by Jack & Heintz, Inc., Box 6719, Cleveland 1, Ohio. Called Trigger Lock, couplings made of aluminum are designed for use with gaseous oxygen and nitrogen, liquid nitrogen, hydrogen peroxide, inhibited red fuming nitric acid, hydrogen tetroxide, jet fuels, hydraulic fluids, exotic power fluids, oil, steam, water and refrigerants. Size ¼ through one inch are proof rated at 4500 psi and burst at 7500 psi. Size 1¼ inch is proof rated at 900 psi with burst at 1500 psi.

Metals, Exotic

Bellows Expansion joints made of titanium are produced by Tube Turns Division of Chemetron Corporation, 224 E. Broadway, Louisville 1, Ky. Joints (18-inch) were made for a chemical processing application in which chlorides in a steam line had been causing stress corrision cracking that limited the life of stainless steel bellows to 30 days with frequent down-time for replacements.

•

Boiler slag is purchased from power companies now instead of the companies having to pay for the slag being hauled away. R. S. Norris & Associates, Larchmont, N. Y., is buying boiler slag at so much per ton, depending upon the vanadium content. Fuel additives containing magnesium, calcium, silicon, etc., have proved helpful in minimizing corrosion in oil fired boilers and in changing the nature of the deposits so that they are more easily removed.

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Non-Metallics

Flexible rubber pipe for use on piping installations with operating temperature ranges to 250 F has been developed by General Rubber Corporation, 67. Summit St., Tenafly, N. J. It is designed for use in buildings having dual heating-cooling systems. Pipe sizes to 12-inch diameters are available in standard lengths to 9 feet.

•

Booklet on silicones containing recent and advanced information is available from Silicones Division of Union Carbide Corp., 270 Park Ave., New York 17, N. Y. Included descriptions of the many uses of silicones for consumer and industrial products and gives properties of silicone fluids, resins, rubber compounds, water repellents, anti-foams and emulsions and their adaptability for use in aviation, automotive, chemical, electronic, rubber, paint, paper, textile, glass, metal working and other industries.

Plastics

Pipe and pipe fittings clad with chlorinated polyether resin to carry corrosive fluids or to withstand corrosive atmospheres are available from York Industrial Plastics, Inc., R. D. 5, York, Pa. Schedule 40 steel pipe and cast iron fittings are clad inside and out with Penton, a polyether resin developed by Hercules Powder Co., Wilmington, Del.

•

Physical, electrical, chemical and optical properties of nine thermoplastic materials are given in a table available from Cadillac Plastic & Chemical Co., 15111 Second Ave., Detroit 3, Mich. Materials covered are acrylics, acetate, butyrate, Teflon and Kel-F fluorocarbons, nylon, polyethylene and vinyls.

•

Lightwall steel conduit armoured with a plastic coating has been used to protect wiring from sulfur dioxide fumes for more than a decade in a large starch plant. The fumes destroyed standard weight galvanized conduit in less than three months, according to the conduit fabricator, Republic Steel Corp., 224 East 131st St., Cleveland 8, Ohio. Polyethylene plastic is extruded directly over the galvanized tubing raceway.

Pumps

A compact, lightweight airless pump designed to give the advantage of airless spray to medium production operations has been developed by DeVilbiss Company, Toledo 1, Ohio. The unit weighs 17 pounds and has a five-gallon capacity pump.

•

Impervious graphite Type F centrifugal pumps are described in a bulletin available from National Carbon Company, Division of Union Carbide Corp., 270 Park Ave., New York 17, N. Y. Four basic pump sizes with capacities to 140 gpm and heads to 67 feet are described.

Switches

Aluminum, explosion-proof, four circuit rotary limit switches are being made by Gemco Electroc Company, 25685 West Eight Mile Road, Detroit 40, Mich. Constructed of light weight cast aluminum to minimize corrosion and to prevent sparking, the switches are designed to meet NEMA specifications.

An all-rubber push button station for oil-tight, heavy duty service is described in a bulletin available from Joy Manufacturing Company, Electrical Products Division, 1201 Macklind Ave., St. Louis 10, Mo. The switch is recommended for all two-button applications such as start-stop, on-off, up-down, etc., and is claimed to be corrosion, dust and moisture proof.

Valves

An air operated throttling valve with a plastic diaphragm and a borosilicate glass body is being marketed by Porter Engineering Co., Hatfield, Pa., in straight through and angle types to fit 1, 1½ and 2-inch pipe. The valve operates on standard 3 to 15 psig instrument air and can be used with metal or glass pipe. It is recommended for handling all liquids except hydrofluoric acid and hot alkalies.

•

PVC dispenser valves designed to eliminate contamination by metals pick-up in demineralized and distilled water systems have been developed by Jobar Equipment Co., P. O. Box 114, South Orange, N. J. These dispensers can be used to handle many chemical corrosives and can be installed anywhere in the laboratory or production facility, according to the manufacturer.

Thermophysical Symposium

The Second Symposium on Thermophysical Properties will be held January 24-26, 1962, at Princeton, N. J., sponsored by the Heat Transfer Division of ASME. Additional information can be obtained from Eric F. Lype, Thompson Ramo Wooldridge, 23555 Euclid Ave., Cleveland, Ohio.

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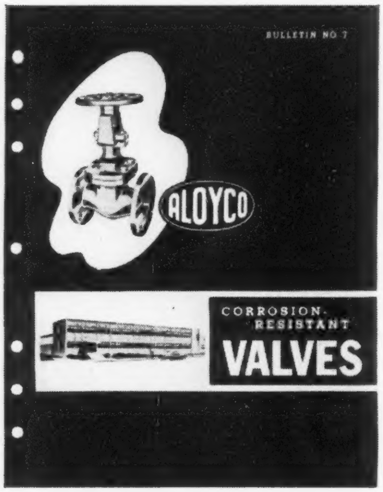

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MEN in the NEWS

Gordon W. Towell has joined the staff of Shell Development Company's Research Center at Emeryville, Cal.

•

Ken C. Tucker has been named assistant sales manager of Oakite Products, Inc., 19 Rector St., New York, N.Y.

•

Richard V. Whitty, Jr., has been named district manager of the Tulsa, Okla., oilfield division office of Hagan Chemicals & Controls, Inc., Hagan Center, Pittsburgh 30, Pa.

•

John L. Wilson has been appointed manager of the newly created production division of filament-wound, reinforced plastic parts of Taylor Fibre Co., Norristown, Pa.

•

NACE Member **Sidney Sussman** has been named technical director of Water Service Laboratories, Inc., 615 West 131st St., New York 27, N. Y.

•

John Ross has been named manager of research and development of Metals and Controls, Inc., 34 Forest St., Attleboro, Mass., a division of Texas Instruments Inc.

•

NACE Member **Ralph F. Strigle, Jr.,** has joined the process equipment division of U. S. Stoneware Company, Akron 9, Ohio, as technical sales representative.

•

Dwight F. Partell has been appointed district manager of Pittsburgh Corning Corporation's office at 18 Camino Sobrante, Orinda, Cal.

•

NACE Member **Joseph R. Robinson** has been appointed Chicago district sales manager for Hill Hubbell Company, 3091 Mayfield Rd., Cleveland 18, Ohio.

•

Donald F. Limburg has been appointed service metallurgist for Jones & Laughlin Steel Corporation's stainless and strip division, Detroit 34, Michigan.

•

William V. Kahler, president of Illinois Bell Telephone Company, 212 W. Washington, Chicago 6, Ill., has received the 1961 Washington Award for distinguished leadership in business and civic affairs and for service to education and humanity. The award is administered by the Western Society of Engineers, 84 E. Randolph St., Chicago, Ill.

•

Howard H. Hoekje has been appointed manager of applied inorganic research for Pittsburgh Plate Glass Company's Chemical Division at Barberton, Ohio.

•

Two NACE members have been promoted in the tubular sales division of Tube-Kote, Inc., 2727 Holmes Rd., Houston, Texas. They are **A. A. Prats, Jr.,** who has been promoted to sales manager of the Gulf Coast division and **Grady Howell,** promoted to sales manager of the northern division, including North Louisiana, North Texas, Oklahoma and Arkansas.

•

Dewey E. Holcombe has been appointed director of field sales for Pfaudler Co., division of Pfaudler Permutit, Inc., Rochester 3, N. Y.

•

NACE Member **J. E. Himmelrich** has been appointed sales manager for pipeline coatings of the protective coatings division of Pittsburgh Chemical Company, Grant Bldg., Pittsburgh 19, Pa. NACE Member **W. D. Moore** has been named assistant to Mr. Himmelrich.

•

NACE Member **Donald W. McCabe** has been named eastern regional sales manager for Polyken Protective Coatings of the Kendall Company's Polyken Sales Division, 309 West Jackson Blvd., Chicago 6, Ill. He will replace **William J. Flaherty,** who has been assigned to a newly created position of manager of the company's Houston, Texas, office.

•

James P. Eagan has joined the stainless and strip division of Jones & Laughlin Steel Corporation, Detroit 34, Mich., as metallographer.

•

John Durham has been named manager of the New York sales district for Garlock Inc., Palmyra, N. Y. **Neal Monger** has been promoted to replace Mr. Durham as manager of the Denver district.

•

Don J. Dicktel has joined the Bruner Corporation, 4767 N. 32nd St., Milwaukee 9, Wis., as a product engineer.

•

NACE Member **Gale Carroll** has been promoted to executive vice president of Tube Process Corp., 6900 Katy Rd., Houston, Texas. J. Hood was elected president of the corporation.

•

Henry A. Denny has been elected vice president and general manager of the engineering and construction division of Koppers Company, Inc., Pittsburgh 19, Pa. **Chester E. Brown** was elected to the same position for the tar products division. **John H. Redmond** was appointed vice president and marketing manager in the tar products division.

•

Uriah M. Brist has been appointed manufacturing superintendent of the Fredericksburg, Va., cellophane plant of American Viscose Corp., 1617 Pennylsvania Blvd., Philadelphia 3, Pa.

•

NACE Member **George O. Rudkin, Jr.,** and **William C. Griffin** have been named associate directors of chemical product development for Atlas Powder Company, Wilmington 99, Del. **Walter H. C. Ruggeberg** has been appointed to the newly created position of director of research and development. **John D. Brandner** and **John W. LeMiastre** have been named associate directors of chemical research. **F. Faxon Ogden** has been appointed research and development planning advisor.

•

Warren H. Dickinson has been appointed product sales manager for pipe protection products of International Division of Minnesota Mining and Manufacturing Co., St. Paul 6, Minn.

•

Howard L. Burpo, Jr., has been appointed regional manager in the midwestern sales region of Union Carbide Plastics Company, Division of Union Carbide Corp., 270 Park Ave., New York 17, N. Y.

•

Two NACE members with Water Service Laboratories, Inc., 614 West 131st St., New York 27, N. Y., have been appointed to new positions. **Walter P. Thurber** has been elected assistant vice president of engineering, and **James B. Fullman** has been appointed chief engineer.

TECHNICAL COMMITTEE ACTIVITIES

Battle Appointed Technical Practices Committee Chairman

Jack L. Battle, senior supervising petroleum engineer with Humble Oil & Refining Co., Houston, Texas, has been appointed chairman of the NACE Technical Practices Committee for a two-year term. He replaces T. J. Maitland of American Telephone and Telegraph Co., New York City.

Battle

One of the first 200 members of NACE, Mr. Battle has been active in all levels of the association's work. He has served as vice chairman of the Technical Practices Committee, Chairman of Technical Unit Committee T-1 on Corrosion in Oil and Gas Well Equipment and has been a member of several technical committees.

His work at Humble deals primarily with corrosion problems including salt water disposal, fluid handling and separation.

He received his education at Southern Methodist University and University of Houston. In addition to his NACE membership, he holds membership in API, AIME and ACS.

As chairman of the Technical Practices Committee, Mr. Battle will be an ex-officio member of the NACE Board of Directors, to which he will make recommendations from his committee for changes so that the aims of the technical committees can be achieved more effectively.

The Technical Practices Committee coordinates the functioning of all NACE technical committees. It establishes procedures necessary for orderly business conduct of the committees and advises on matters of policy and planning which affect the technical committees.

Nace Technical Reports

STRESS CORROSION CRACKING. 15 papers reprinted from Corrosion. Pub. 59-4. Postpaid, Per copy $5

HIGH PURITY WATER CORROSION. Sixteen papers reprinted from Corrosion. Pub. 59-3. Postpaid, per copy $3

1957-58 NACE TECHNICAL REPORTS. 38 reports reprinted from Corrosion. Includes some in Pub. 59-2 and 3 above. Postpaid, per copy . . $6.50

Remittances must accompany all orders for literature the aggregate cost of which is less than $5. Orders of value greater than $5 will be invoiced if requested. Send orders to National Association of Corrosion Engineers, 1061 M & M Bldg., Houston, Texas. Add 65c per package to the prices given above for Book Post Registry to all addresses outside the United States, Canada and Mexico.

Thermoelectric Generators May Supply Impressed Current Power

Thermoelectric generators as sources of impressed current for cathodic protection systems may be produced eventually for less than **$100** with a **300-watt** capacity, according to a report of Task Group T-2B-1 (Sources of Impressed Current for Anodes) given at the Dallas meeting of NACE Technical Unit Committee T-2B on Anodes for Impressed Currents.

Thermoelectric generators have good possibilities for applications on cathodic protection installations, one company representative stated. A trial installation has a d-c rating of 3 amperes at 3 volts, using methane as the heat source. The low-voltage, high-current thermocouples used in the trial device can generate a potential of 140 millivolts at a temperature range of 1000 F. Thermocouples can be produced with capacities to 50 amperes, it was stated.

Efficiency of the trial thermoelectric unit was about 3½ percent with a 10-pound pressure required in the gas supply.

Service life from such thermoelectric devices can be expected to be indefinite if the hot-side temperature does not exceed about 1200 F, according to the discussions reported in task group minutes.

Deep Anode Task Group

Another task group report given at the T-2B meeting was from T-2B-6 on Deep Anodes. A deep anode ground bed installed in Mississippi back in 1941 is still performing satisfactorily for cathodic protection of a gas distribution system. This deep bed used an abandoned 8-inch water well casing, about 600 feet deep. Deep ground beds have been used on tank farm pipeline systems with a protective current spread that could be detected for distances of 35 and 40 miles in each direction on the pipeline.

High Silicon Cast Iron Anode Report

Concrete encased steel anodes have proved unsatisfactory, according to tests discussed in another task group report given at the T-2B meeting. Initial resistance of the concrete encased anode increased until cracking of the concrete occurred, after which the anode-to-earth resistance dropped, according to the Task Group T-2B-4 (Use of High Silicon Cast Iron for Anodes).

Current density of 20 milliamperes per square foot passing through concrete will cause cracking, according to the discussion. No damage of concrete was observed at 2.0 milliamperes per square foot. When reinforcing steel is maintained as a cathode at current density of 5.0 ma per square foot or more, the concrete will soften around the steel.

Pretreatment Advantages Given For Steel Exchanger Tubes

A case history showing the advantage of pretreatment of steel heat exchanger tubes was presented at the Milwaukee meeting of Task Group T-5C-2 on Corrosion by Cooling Water—North Central Region, held during the North Central Region Conference.

The tubes in the catalytic cracking unit Exchanger were fabricated primarily of mild steel with a few admiralty bundles included in the system. All bundles were pretreated on the water side except for one heat exchanger which was not pretreated to study the benefits of pretreatment.

Four exchangers were opened after hydrotesting and before the unit went on stream for the first time. Numerous tubercles and a coating of rust on the metal surfaces in the channel and channel head section of the untreated exchanger were apparent. The treated bundles were coated smoothly with inhibitor.

This case history was described as an indication that pretreatment is a necessary step to obtain life and trouble-free operation of new equipment.

The task group also heard a report on the data collected from a small group of questionnaires sent primarily to water treatment manufacturers. The survey was to determine methods used for measurement of corrosion in cooling water systems and to determine treatment efficiency.

A committee was appointed to prepare a manual on cooling water treatment to include the following: (1) fundamental aims in establishing a treating program, (2) cooling water system control requirements, (3) recommended operating procedures and (4) chemical testing methods.

Ohio Corrosion Committee Holds Management Meeting

The Columbus and Central Ohio Committee on Corrosion, affiliated with NACE Technical Committee T-7B, held its annual meeting for management in January. Purpose of the meeting was to acquaint management personnel with the problems confronting corrosion engineers and to show the operation and functions of a corrosion coordinating committee.

Special cases were included in the talks given by guest speakers to show the functions of corrosion coordinating committees throughout the country.

●

Sale of books in the United States totaled $630 million in 1959.

Burton

Caldwell

Hoy

Rowe

Four Group Committee Chairmen Appointed for 1961-63 Terms

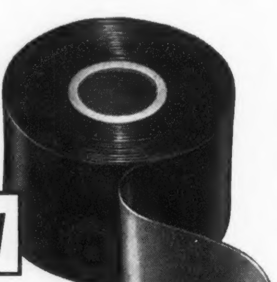
Chairmen for four NACE technical group committees have been appointed for two year terms extending through March, 1963. They are to head Group Committees T-1 (Corrosion in Oil and Gas Well Equipment), T-3 (General), T-5 (Corrosion Problems in the Process Industries) and T-7 (Corrosion Coordinating Committee).

T-1 chairman is J. A. Caldwell of the Production Research Division of Humble Oil & Refining Co., Houston, Texas. Active in NACE since 1946, he has been vice chairman of T-1, chairman of the South Central Region, chairman of the Houston Section and currently is serving as director of the South Central Region.

T-3 chairman is Leonard C. Rowe, senior research chemist in the Chemistry Department, Research Staff, General Motors Corp., Warren, Mich. He has been a member of several NACE technical committees and has been active in the Detroit Section, of which he currently is vice chairman. He also is CORROSION Magazine correspondent for the transportation industry.

T-5 chairman is W. H. Burton, General Chemical Division, Allied Chemical Corp., Camden, N. J. Active in NACE technical committee work since 1953, he has been vice-chairman of T-5 the past year and has been a member of several unit committees and task groups.

T-7 chairman is Paul C. Hoy, Hayton Power & Light Co., Dayton, Ohio. He has been active in NACE work, has served as chairman of T-7B and has been vice chairman of T-7 during the past year.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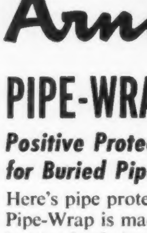
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Deep Anode Ground Bed Advantages Given In T-2B-6 Report

Advantages, disadvantages and ten methods of installing deep anode ground beds are discussed in the first interim report submitted to members of Technical Unit Committee T-2B (Anodes for Impressed Currents) by Task Group T-2B-6 on Deep Anodes.

Advantages discussed in the report include installation of deep ground beds on the usual limited pipeline right-of-way with no need for acquiring installation rights on adjacent lands as is usually necessary with conventional ground beds.

This first interim report consists of an informational summary of the various methods that have been used to install deep anodes. This information was compiled from material submitted by subcommittee members and from literature references.

A second report submitted to T-2B committee members was from Task Group T-2B-7 (Lead Anodes). This report was designed to acquaint committee members with details concerning the use of lead alloy anodes in various electrolytes as distinct from system considerations. The report explains how lead alloys differ from other anodes as current sources and gives some indication of where lead anodes are used successfully and where they might be used.

Effect of Inhibitors On Sulfuric Acid Alkylation Discussed

The effect of inhibitors in neutralization and washing operations for sulfuric acid alkylation was one of the discussion topics at the Tulsa meeting of Technical Group Committee T-8 held during the South Central Region Conference.

Severe corrosion in the reactor effluent lines and isobutane tower reboilers after service of two to six months in three alkylation units was described by one committee member. As a control on acid carryover from the caustic wash drum, corrosion meter probes were used between the caustic and water wash drums. These probes have been effective in identifying acid carryover. To control ester corrosion in the reboilers, an oil soluble inhibitor was injected at one quart per thousand barrels at the caustic drum outlet.

This plant is operating on a two-year inspection cycle with no plugged bundles. Corrosion rate has been lowered from 35 to 40 mpy to 2 to 6 mpy.

Another member reported that corrosion of total alkylation pumps had cut service life to 9 or 10 months. Inhibitor injected ahead of pumps extended pump life to three or more years. Injection rates of ¼ and ½ quarts per thousand barrels were ineffective, but the rate of one or two quarts per thousand barrels gave the same protection.

Hastelloy 3 eductors gave four years' service in contrast to carbon steel which lasted only a few days and epoxy lined steel which lasted a few weeks, according to another committee member. Teflon sleeves have given over 6 months' service. Saran lined eductors have reportedly given 6 years' service, according to the discussion.

Other discussion topics at the T-8 meeting included exchanger and condenser corrosion, waterside corrosion problems, corrosion in reforming and desulfurization units, metallurgy phenomena, plastics in the refining industry and high temperature furnace tube corrosion.

As part of its business meeting, Committee T-8 heard a report from its Task Group T-8A on Corrosion Control in Chemical Cleaning. A status report was made on an inhibitor test program.

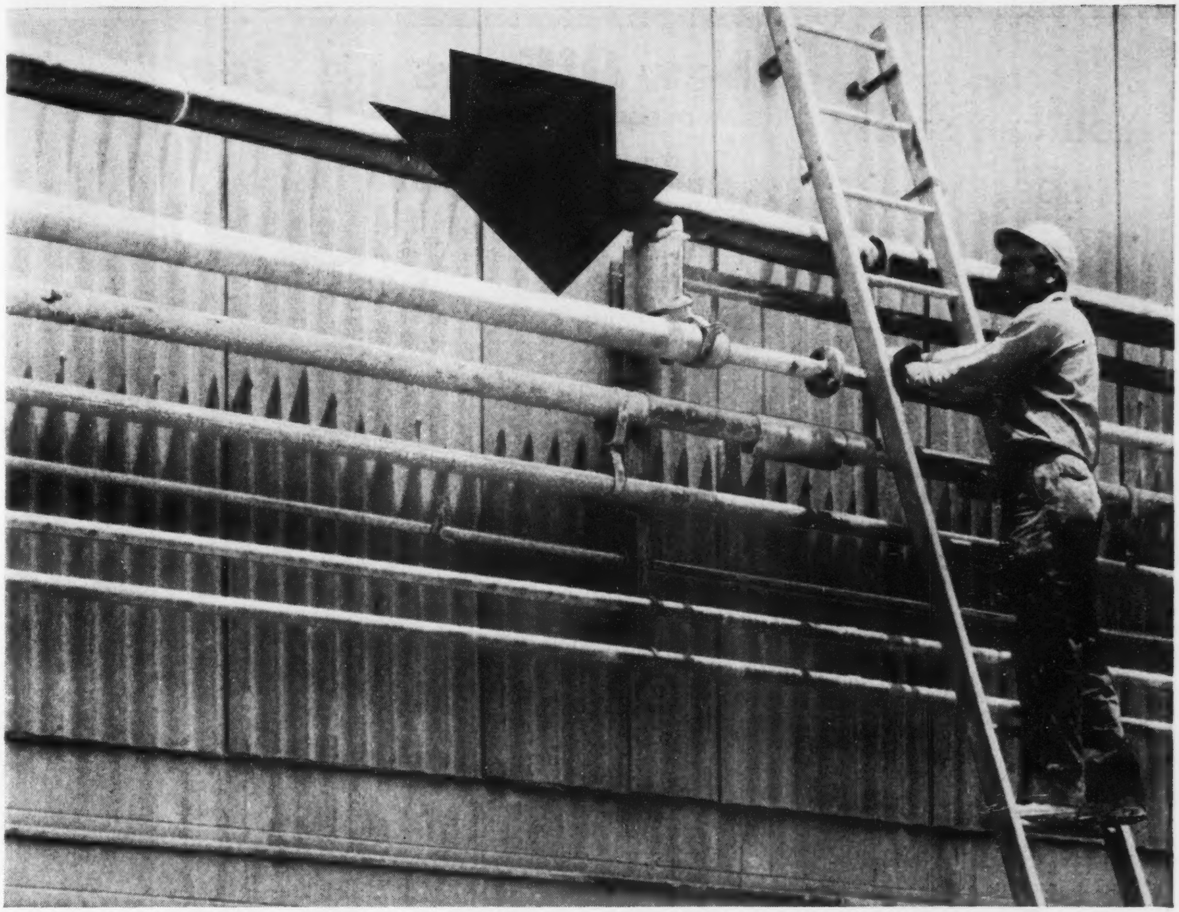
Two Task Groups Formed By Unit Committee T-1E

Two task groups have been established by Technical Unit Committee T-1E (Cathodic Protection of Oil Field Equipment) to study the application and evaluation of cathodic protection as a corrosion control method for all types of oil field equipment—both surface and subsurface. The two groups will be Task Group T-1E-1 (Cathodic Protection of Surface Equipment) and Task Group T-1E-2 (Cathodic Protection of Subsurface Equipment).

Chairman for T-1E-1 is C. O. Smith of the Carter Division of Humble Oil & Refining Co., Tulsa, Okla. The other chairman is to be selected.

This meeting of T-1E was held at Tulsa, Okla., during the South Central Region Conference and was its first

(Continued on Page 46)



Resistant Ni-o-nel alloy quadruples service life of line handling "wet process" phosphoric acid

Lasts four times longer. Resistant Ni-o-nel* nickel-iron-chromium alloy has outlasted the best material previously used for this pipe, by a margin of four to one. This pipe handles "wet process" phosphoric acid, at Virginia-Carolina Chemical Company's Nichols, Florida plant. Acid concentration is 55% P_2O_5. Temperature is 200°F. Acid contains small amounts of dissolved impurities—calcium sulfate, fluorides, iron and aluminum.

Experience at other plants:

Ni-o-nel alloy cylinder and baffle plate in submerged combustion evaporators. Concentrate 29% to 54% P_2O_5. Impurities include 1-3% sulfuric acid, 1-2% fluorides, 3-20% suspended solids. Temperature range is 220°-280°F.

(Ni-o-nel alloy has stepped up the life of these parts 3 to 5 times.)

Ni-o-nel alloy cone sections of venturi scrubbers. Used in producing 48-50% phosphoric acid from elemental phosphorus. Temperature range of 175-200°F. High agitation.

(Ni-o-nel alloy metal losses have reached a maximum of 0.0065 ipy in pure runs. In runs containing a high percentage of fluorides and other impurities the high was 0.014 ipy.)

In pure phosphoric acid, Ni-o-nel alloy resists all concentrations and temperatures up to and including a boiling 85% solution. It is successful, for example, in equipment polymerizing hydrocarbons by phosphoric acid catalysis. Also, in the heating coils used in applying phosphate coatings to steel.

Have you a phosphoric acid corrosion problem? If so, send for a copy of our Technical Bulletin T-37, "Engineering Properties of Ni-o-nel." This informative publication contains practical information about corrosion resistance, fabricating procedures, welding, available mill forms, mechanical properties, and physical constants.

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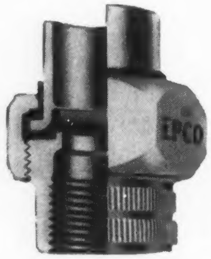

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T-5 Vice Chairman

C. P. Dillon, Union Carbide Chemicals Co., South Charleston, W. Va.

T-5B Officers

Chairman D. W. McDowell, Jr., International Nickel Co., Inc., New York, N. Y., and Vice Chairman J. R. Myers, Wright Patterson Air Force Base, Ohio.

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Chairman M. W. Belue, Jr., Champion Paper & Fibre Co., Hamilton, Ohio, and Vice Chairman S. W. McIlrath, Diamond Alkali Co., Cleveland, Ohio.

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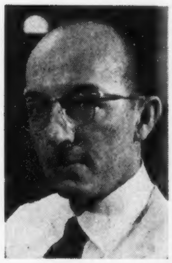
Simmons

Effects of Handling Practices Discussed At T-1F Tulsa Meeting

Handling practices affect the corrosion characteristics of tubular goods, but these effects are other than general metal loss, according to statements made at the Tulsa meeting of Technical Unit Committee T-1F on Metallurgy of Oil Field Equipment, held during the South Central Region Conference.

Failures by sulfide stress cracking and stress corrosion cracking have been frequent enough to arouse some concern about the effect of handling practices.

Hydrogen sulfide has several effects on oil well tubing, according to the discussion. The first is the distinct effect of hydrogen derived from corrosion reactions with fluids containing hydrogen sulfide. On soft steels, this hydrogen reduces ductility; on hard steels, this hydrogen can cause spontaneous cracking —called sulfide stress cracking. The exact mechanisms of this has not been clarified completely to-date.

At low concentrations of hydrogen sulfide under some conditions, a third type of damage can occur, commonly known as stress corrosion cracking.

To minimize the effect of hydrogen sulfides in oil well fluids, six precautions should be observed, according to the discussion at the task group meeting: (1) Steels should be selected to have physical properties consistent with serevity of the environment. Maximum hardness and tensile strength and degree of cold deformation such as straightening and handling dents should be controlled. (2) Full contact slips and tongs should be used for minimum identation. (3) Consideration should be given to incidental operations such as acidizing which may aggravate the hydrogen effects. (4) Kinked tubing should be rejected. (5) Spot inspection for stress corrosion cracking should be made when tubing in suspected environments is removed from the wells. (6) Stresses should be kept low if possible.

T-1E Task Groups—

(Continued From Page 44)

meeting since the reorganization of T-1.

The committee also heard a review of case histories of cathodic protection stray currents under investigation in California oil fields. Conclusions drawn from the case histories' casing potential data, stray current on oil well casings caused by cathodic protection seems to be small and about the same rate as naturally occurring galvanic currents. The resulting damage is insignificant if the stray current leaves the casing uniformly.

•

The 6th Annual Appalachian Underground Corrosion Short Course will be held June 6-8 at the University of West Virginia, Morgantown.

HOW CAN DU PONT PAINTS CUT YOUR MAINTENANCE COSTS 50%?

Ask Larry Schmeig. If you're caught in the cost-price squeeze these days, you'll welcome the assistance of a man like Larry. A member of Du Pont's special Technical Service Group, he's trained specifically to help you solve maintenance painting problems and improve profits. As Larry puts it:

"When you're talking economy in maintenance painting, initial cost per gallon is a small part of the total picture. Much more important, in terms of real savings, are maximum efficiency of application and length of time the job lasts. As a matter of fact, in a recent 7-year period, we were able to cut painting costs at Du Pont 50%—for a net maintenance saving of more than $1,000,000 a year!

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Whatever your maintenance problem, there's a *proven* Du Pont finish to help you solve it. And new products are added regularly. Examples: CORLAR(T.M.) Epoxy Chemical-Resistant Finishes, for dependable protection against acid fumes, alkalies and solvents; IMLAR(T.M.) Vinyl Coatings, for *maximum* resistance to severe acid and corrosive conditions. For detailed information, clip and mail the coupon below. Or see the Du Pont Technical Representative in your area.

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NACE NEWS

NACE Member Trains Coating Personnel For Indian Government

NACE Member Charles B. Masin has been training Indian personnel in specialized protective coating applications and inspection procedures on the Bhakra Dam, one of the world's largest concrete dams. The structure now is nearing completion in the Himalayan foothills of the Punjab on a tributary of the Indus River.

On the Bhakra Dam, hot coal-tar enamel is being applied to the interior of five 15-foot diameter steel penstock. An indigenous hot coar-tar enamel is being service tested in several river outlet pipes. Trashracks have been coated by dipping in hot coal-tar pitch. Gates and other ferrous metalwork are being given cold applied protective coating systems.

Mr. Masin was employed in April, 1959, by the Indian Government as supervisor of painting and coal-tar enameling on the dam project. Formerly he was associated with the U.S. Bureau of Reclamation as materials engineer on protective coatings for dams, power plants and irrigation structures.

Western Region

San Francisco Bay Area Section had George E. Moller of Union Oil Company of California for its guest speaker at the February 14 meeting. His topic was zinc and lead-silver anodes as a method of solving six refinery corrosion problems.

•

The 1961 Western Region Conference will be October 4-6 at the Hotel Multnomah, Portland, Oregon.

Boston Section Short Course June 20-22 Program Given

Complete program for the June 20-22 Corrosion Short Course sponsored by the Greater Boston Section of NACE is given below. The three-day course is planned to give fundamental training in corrosion and will cover the causes of corrosion, construction materials, coatings, cathodic protection and practical solutions to corrosion problems.

Registration fee for the course will be $15. A tour is being planned for ladies who may be accompanying registrants.

Each lecture session will be followed by a discussion period, according to short course committee chairman W. S. Sanders. J. R. Sarnosky is program chairman.

Tuesday, June 20

Need for Corrosion Education, by M. Glover of Glover Coating.

Need for Corrosion Engineering, by Robert Zinn of A. D. Little Company.

Fundamental Science of Corrosion, by H. D. Gates of Massachusetts Institute of Technology.

Film: "Corrosion in Action," produced by International Nickel Co., Inc.

Wednesday, June 21

Materials of Construction—Iron and Nickel Base Alloys, by W. Z. Friend of International Nickel Co., Inc.

Materials of Construction—Aluminum and Copper Alloys, by Wilson Lynes of Revere Brass & Copper Co.

Organic Coatings—Properties, by R. Gackenbach of American Cyanmid.

Coatings and Linings—Applications, by H. Foelsch of Lithcote Corp.

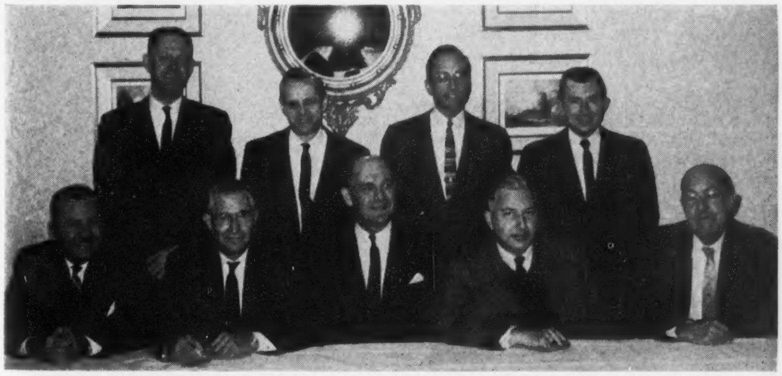
Thursday, June 22

Principles of Cathodic Protection, by T. P. May, International Nickel Co., Inc.

Applications of Cathodic Protection, by A. W. Peabody of Ebasco Services.

Designing to Prevent Corrosion, by S. K. Cobern of U.S. Steel Corp.

Practical Solutions to Corrosion Problems, by J. H. Peacock, of Duriron Company.



PITTSBURGH SECTION honored its past chairmen at the February 2 meeting. Nine of the 11 past chairmen are shown above. Seated left to right are F. E. Costanzo (1953-54), N. P. Peifer, Sr. (1952-53), Russell H. Coe (1949-50), J. M. Bialosky (1951-52) and V. V. Kendall (1950-51). Standing left to right are Wayne Binger (1954-55), R. W. Maier (1959-60), L. G. Royston (1956-57) and S. H. Kalin (1958-59).

Northeast Region

Baltimore-Washington Section heard a panel of three men discuss their corrosion problems encountered in municipal water handling in the District of Columbia. They were D. V. and J. W. Krasauskas and Allan B. Fay. They explained that the District has no separate department to handle corrosion problems; consequently each department head tries to solve his own problems by trial and error.

•

Kanawha Valley Section heard Warren Immel of Tennessee Gas Pipeline Company speak on cathodic protection in plant yards using distributed ground beds at the January 19 meeting.

At the March 23 section meeting, J. V. Petrocelli of International Nickel Co., Inc., Bayonne, N. J., spoke on electrochemical reactions.

•

Philadelphia Section had a panel discussion on coatings and surface preparation at the February 7 meeting. Panel members were Kenneth Lefevre of Metalweld, Inc., Joseph M. Walters of Electro Chemical Engineering & Mfg., and Edward Brink, panel moderator, of American Viscose Corporation.

•

Greater Boston Section has scheduled Douglas E. Noll to speak on corrosion in boilers and condensate lines at the April 12 meeting, which will be open to the Plant Engineers Club. New section officers also will be elected.

Albert P. Richards, president of William F. Clapp Laboratories, spoke on biological influences on deterioration of materials in marine environments at the February 15 meeting, attended by 38 members and guests.

•

Wilmington Section has planned its programs for March, May, September and November as follows:

March 22: E. D. Edminsten of Fibercast Company will speak on applications of reinforced plastic pipe.

May 24: Guest speaker will be W. Z. Friend of International Nickel Co., Inc.

September 20: C. E. Fox of Dow Industrial Service will speak on chemical cleaning.

November 15: James E. Parkinson of the U.S. Navy Yard will speak on protective coatings.

•

The 1961 Northeast Region Conference will be Oct. 20-Nov. 2 at the Hotel Statler, New York City.

Almost 2000 Registered at Buffalo Conference

Attendance Good At 53 Technical Committee Meetings

Attendance was close to the 2000 mark at the 17th Annual NACE Conference last month in Buffalo, N. Y. Total registration was 1923. Included were representatives from foreign countries: Mexico, Canada and Sweden. The 50th state, Hawaii, also was represented.

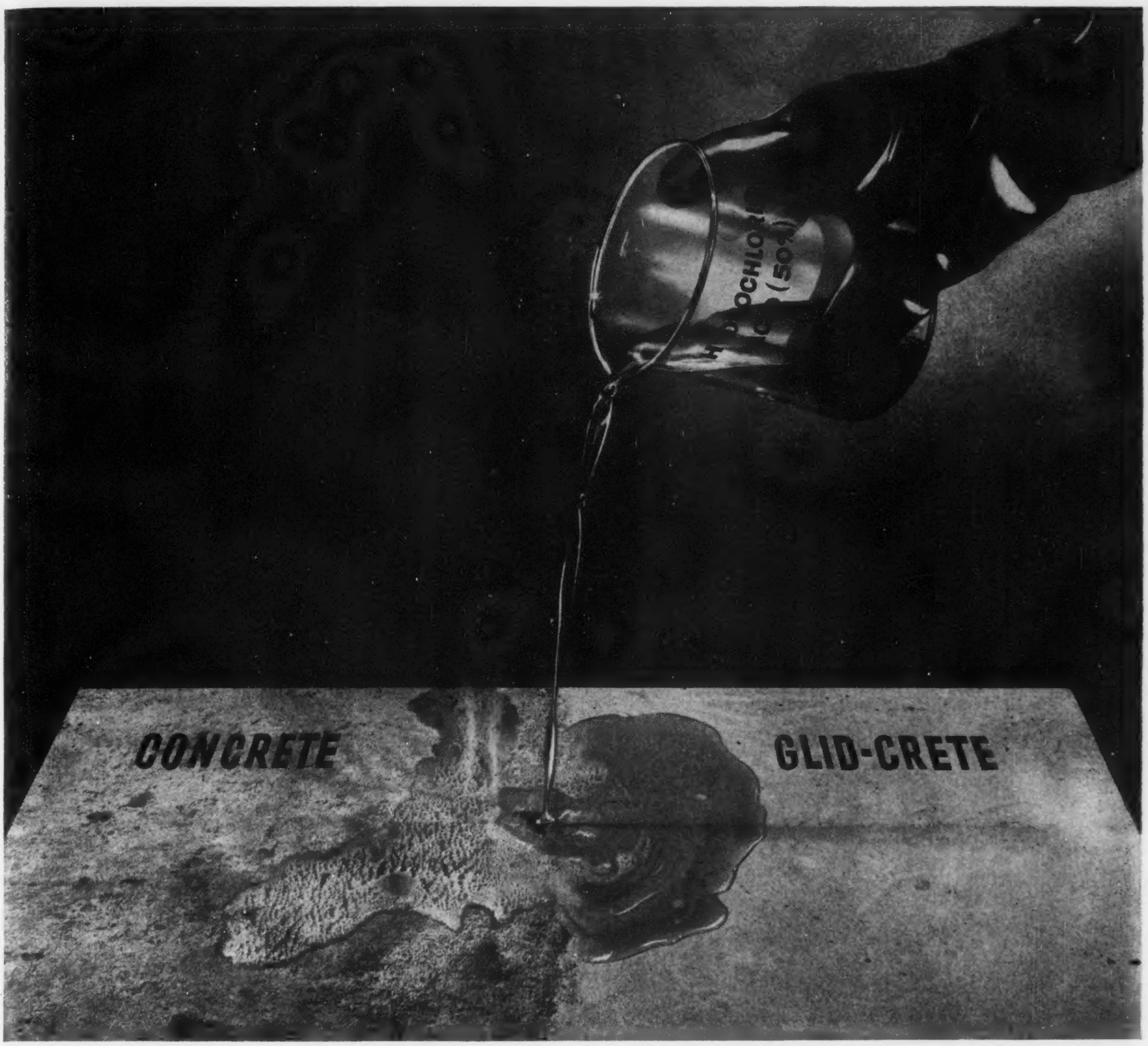
The high attendance was attributed in part to the fact that the weather in Buffalo was good—no airline flights were cancelled because of weather either before or during the conference.

Attendance was good at the 53 technical committee meetings and all the symposium sessions on the technical program. Exhibitors at the 1961 Corrosion Show stated that a high level of interest was shown by registrants in the 101 booths where the latest in corrosion control equipment, supplies, processes and techniques were displayed.

Unusual interest was shown by the Buffalo newspapers which covered the conference activities with daily stories and photographs. The two local television stations also covered the conference: one televised an interview with Frank N. Speller; the other station aired motion picture coverage of the Corrosion Show.

Photographic coverage of the conference activities including the fellowship hour, banquet, technical committee meetings, symposia and the Corrosion Show appears on Pages 54-64 of this issue.

Top photograph shows a part of the registrants at the annual fellowship hour at which old acquaintances are renewed and new ones made. Photographs at right were made at the registration desk. Although almost 2000 people were registered, long waiting lines were avoided by the efficiency of the ladies at the registration desk.



Even strong acid can't etch concrete floor coated with NEW GLID-CRETE

Floors "floored" by acids and chemicals? Here's the answer . . .

New GLID-CRETE, based on a catalyzed epoxy resin, used to patch or resurface concrete floors was given the acid test. A 50% solution of hydrochloric acid was poured on bare concrete and on concrete topped with ⅛" of GLID-CRETE. The concrete was badly etched. GLID-CRETE was unharmed—and will stay that way!

Easy to apply, GLID-CRETE is six times more abrasion and impact resistant than concrete. It offers many outstanding advantages in the maintenance, repair and construction of masonry floors and structures.

GLID-CRETE can save you money. Time too. Write today for complete information.

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AZI

The American Hot Dip Galvanizers Association with the cooperation of The American Zinc Institute Announces the $10,000.

Galvanizers' International

Ten Awards of $1,000. Each

AMERICAN HOT DIP GALVANIZERS ASSOCIATION

Ten awards are to be made to entrants selected by the judges. Each award will consist of $1,000 in cash, a suitable medal and a Certificate of Achievement.

This is not a contest—it is a search for new ideas. ■ *Your entry, therefore,* will not be judged against others, but solely on its merit and value in developing new applications and markets for Hot Dip Galvanizing. ■ If your idea, in the opinion of the judges, is of practical value to the industry, you will be cited for an award—promptly. ■ Because the Hot Dip Galvanizing Industry is anxious to receive ideas of this type, the judges reserve the right to present *more* than 10 awards, if the entries warrant.

CONDITIONS ■ Anyone in the world (except members of the American Hot Dip Galvanizers Association and the American Zinc Institute, and their employees and advertising agencies) may submit one or more entries. ■ Entries will be considered by the judges promptly upon their receipt. No entry received after April 30, 1962 will be considered. ■ The Awards will be made for ideas pertaining to: (a) Applications of Hot Dip Galvanizing to a *new or unusual* field, or; (b) An *improvement* in application in fields where Hot Dip Galvanizing is now being used, or; (c) New methods of *after-treatments* of Hot Dip Galvanized products. ■ Each entry submitted must contain: (a) Description and documentation of application. (b) Case history of the application or process accompanied by photo, drawings, formulae, etc. (c) All technical data needed for the utilization of the idea submitted. (d) Release of the application or idea for general use without payment or royalty other than the $1000 award. ■ The decision of the judges will be final. Award-winning ideas will be retained by the American Hot Dip Galvanizers Association for dissemination throughout industry. Other entries will be returned. ■ No formal entry blank is required but the entry should be accompanied by the name, address and business connection of the individual submitting it. Business firms or corporations may submit entries under their business name, instead of as individuals, if they choose to do so. ■ Entries should be sent to: American Hot Dip Galvanizers Association, Inc., 5225 Manning Place, N.W., Washington 16, D. C. ■ *Note:* For information on galvanizing write to the above address for name and location of the American Hot Dip Galvanizers Association member nearest you.

Awards

FOR ACHIEVEMENTS IN RESEARCH, DEVELOPMENT AND UTILIZATION OF GALVANIZED PRODUCTS

THE JUDGES ■ Dr. Clarence H. Lorig, Technical Director, Battelle Memorial Institute and Past President American Society for Metals. ■ Mr. John R. Daesen, Technical Director, American Hot Dip Galvanizers Association. ■ Mr. John L. Kimberley, Executive Vice President, American Zinc Institute.

STATLER HILTON
1

STATLER HILTON
2

ANNUAL BANQUET

Photograph 1 shows Kenneth G. Compton (left) of Bell Telephone Laboratories, Inc., Murray Hill, N.J., receiving the 1960 Speller Award from NACE Past President Hugh P. Godard of Aluminium Laboratories, Ltd., Kingston, Ontario, Canada. H. R. Copson (at left in Photograph 2) is being presented the 1960 Whitney Award by Mr. Godard. Photograph 3: Mrs. Walter Szymanski, wife of the conference general chairman, has presented Hawaiian leis to the new NACE officers (left to right): President Edward C. Greco of United Gas Corp., Shreveport, La., Treasurer Charles G. Gribble, Jr., of Metal Goods Corporation, Houston, Texas, and Vice President R. McFarland, Jr., of Hills-McCanna Company, Chicago, Ill. Photograph 4 shows Mrs. Szymanski assisted by Mrs. Tom Brinkworth of Buffalo N.Y., presenting the lei to President Greco. Seated at right is Immediate Past President George E. Best of the Manufacturing Chemists' Association, Inc., Washington, D. C.

Hawaii Section Proposed

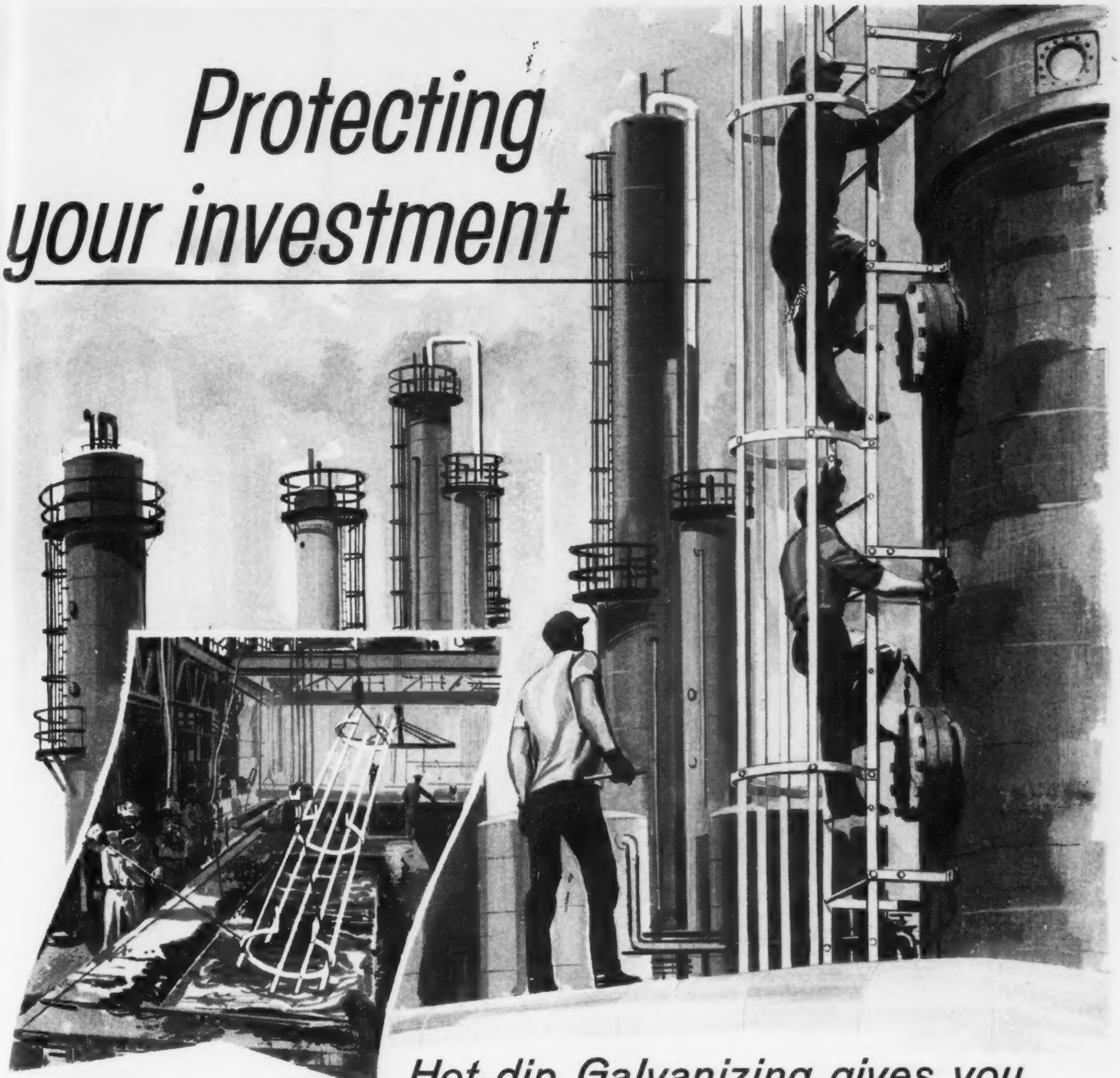
Floral leis flown in from Hawaii were presented to the NACE officers and wives of the local arrangement committee officers at the banquet held during the Buffalo Conference, March 13-17.

The leis were furnished by some of the Hawaiians who are interested in forming an NACE section in the islands, now that Hawaii is a state and since the NACE articles of organization and bylaws have been changed to permit organization of sections in Alaska and Hawaii.

Mrs. Walter Szymanski, wife of the conference general chairman, presented the leis to outgoing NACE President George Best, Edward C. Greco, now president, Vice President R. McFarland, Treasurer Charles C. Gribble, Jr., Ivy Parker, editor of CORROSION and Executive Secretary T. J. Hull.

The presentation was arranged by Harland A. Morley, NACE member from the Pearl Harbor Naval Shipyard, Honolulu, Hawaii.

Protecting your investment



Hot-dip Galvanizing gives you dividends in protection

Nowery J. Smith Co. hot-dip galvanizing gives you *sure* long-term metal protection. Hot-dip galvanizing affords the advantages of complete and uniform zinc coating on complex shapes, inside surfaces and other hard-to-reach areas. Nowery J. Smith Co. has the largest hot-dip galvanizing facilities in the Southwest. Items up to 63-foot structural beams can be hot-dip zinc galvanized at the Company's Houston plant.

Nowery J. Smith Co. also offers sand and shot blasting, metallizing, prime coating and painting, pickling, oiling, polyvinyl chloride PLASTISOL coating and other special protective coatings. We are exclusive in the Southwest for application of Goodyear Rubber Company's PLIOWELD Linings.

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Photograph 5: H. S. Preiser is shown presenting his technical paper on cavitation damage during one of the sessions of the Cathodic Protection Symposium. Seated next to the podium is the symposium chairman, Sidney Tudor of the New York Naval Shipyard.

Photograph 6: NACE Technical Committee T-8A on Chemical Cleaning was one of the 53 technical committee meetings held during the conference.

Photograph 7: Many informal technical discussions continued in the halls between symposia meetings. Shown here (left to right) are R. J. Law of International Nickel Co., Toronto, Canada, Ross McCuaig of Polymer Corporation Limited, Sarnia, Ontario, and W. S. Chisholm.

Photograph 8: Three Canadian registrants at the conference in another informal discussion are (left to right) H. Murray Armstrong of Caproco Corrosion Prevention, Edmonton, Alberta, Walter Morris of Interprovincial Corrosion Control Company, Ltd., Calgary, Alberta, and Joseph Messenger of Mobil Oil of Canada, Ltd., Calgary, Alberta.

$36 worth of Corban helped oil producer save more than $3000 in six months

DOWELL
CORBAN
corrosion
inhibitor

Here is a story that shows how the effective low-cost corrosion protection offered by Corban® helped reduce costs and increase net profits for an oil producer. The well was a pumper in Wayne County, Illinois. The operator was plagued with corrosion-caused sucker rod breakage. Sixteen rod partings had occurred in less than eleven months, including rods in a new string which had been in the well about two months. Service costs alone for the fishing and rod-pulling jobs totaled $1020. This did not include downtime or rod replacement costs.

Dowell recommended treating with Corban 207. Material cost was only $5.40 per month.

At last report, $36 worth of Corban had been used, and there had been no rod failures for six months. Based on past experience, the operator estimated that the $36 worth of Corban had saved him more than $3000.

If you have a corrosion problem, call your Dowell representative. He will help you develop an effective, low-cost treatment program designed to reduce operating costs and increase profits. Dowell services and products are offered from more than 150 offices and stations in the United States, Canada, Venezuela, Argentina, Germany, France and the Sahara area. Dowell, Tulsa 1, Oklahoma.

PRODUCTS FOR THE OIL INDUSTRY **DOWELL**

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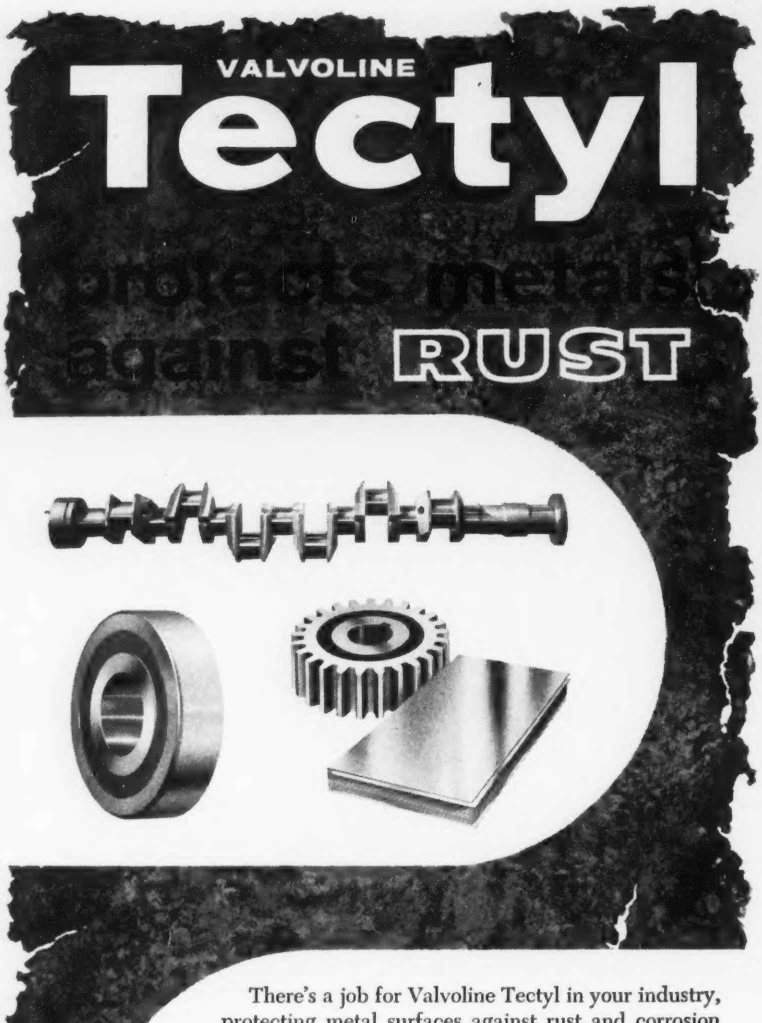
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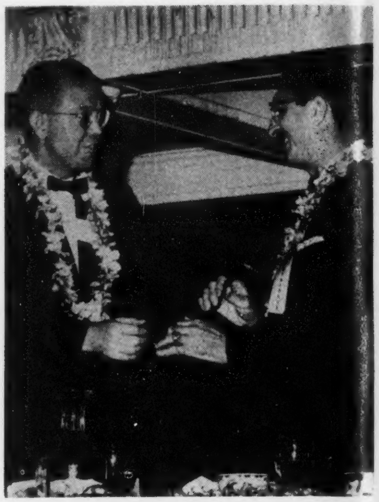
Tectyl RUST PREVENTIVES

VALVOLINE OIL COMPANY, Division of Ashland Oil & Refining Company
Refinery — Freedom, Pennsylvania — Home Office — Ashland, Kentucky



FORMAL PRESENTATION of the gavel to the new president of NACE, Edward C. Greco of United Gas Corporation, Shreveport, La., (at right) by outgoing President George E. Best was made at the banquet. Mr. Best was given a small copy of the gavel with his name engraved on it as a momento of his having served as NACE president for 1960-61.

Greco to Represent NACE At London Conference

E. C. Greco, United Gas Corp. Shreveport, La., NACE president, has been authorized by the association's board of directors to present official greetings from NACE to the First International Congress on Metallic Corrosion in Londen. Mr. Greco, and some 20 other NACE members are scheduled to present papers or participate otherwise in this congress April 10-15.

By-Laws Changed to Permit Sections in Hawaii, Alaska

The NACE board has approved amendments to the association's articles of organization and by-laws which will permit organization of NACE sections in Alaska and Hawaii. The articles previously stipulated that continental North America was the geographical limit for sections.

During the past two years there has been a rapid growth of interest in corrosion control in Hawaii. Members there have been urgently petitioning for recognition. While there is no present indication of substantial interest in Alaska, the association is now in position to organize a section there.

The NACE board also approved changes in the articles whereby an NACE member may choose his section affiliation. This change comes about because of the fact that on the borders of regions, members in an adjacent region often find it convenient to belong to a section in the adjacent region. This also will permit members, by reason of section affiliation, to vote in the region in which their section is located.

•

NACE's 18th Annual Conference and 1962 Corrosion Show will be held March 18-22, 1962, at the Municipal Auditorium in Kansas City, Mo.

In pulp mill hot acid system

Cast nickel stainless steel "Y" valve . . . resists corrosion for 31 years, is still trouble-free

After 31 years of service on a digester relief line handling hot sulphite liquor, steam and hot SO_2 gas, this cast stainless "Y" valve is as good as the day it was installed.

And this cast valve is expected to give many, many more years of trouble-free service . . . despite operating temperatures up to 270°F and operating pressures up to 50 psig.

Long life cast in. To assure a long service life right from the start, the valve was cast in an alloy equivalent to Type CF-8M* (19% Chromium, 10% Nickel, 2.5% Molybdenum).

This nickel-containing casting alloy provides a high degree of corrosion resistance and strength, making it an ideal choice for valves and pumps that must withstand the constant attack of a variety of corrosive materials throughout industry.

An entire family of cast austenitic nickel stainless alloys is available today to give you equipment that will resist corrosive attack, protect your product against contamination, and deliver long, low-maintenance service.

To help you select the right nickel-containing casting alloy, we've prepared a detailed 72-page booklet: "Heat Resistant Castings, Corrosion Resistant Castings . . . Their Engineering Properties and Applications". Just drop us a note requesting Booklet A-266. A copy will be sent to you immediately.

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INCO NICKEL

NICKEL MAKES ALLOYS PERFORM BETTER LONGER

NIAGARA FRONTIER SECTION recognized some of its charter members at a special fellowship hour held during the Buffalo Conference. They are (left to right) Sando Biondolillo of Bell Aircraft Corporation, Buffalo, N.Y., William O. Binder, T. R. B. Watson of Corrosion Service Limited, Toronto, Canada, C. R. Bishop, chairman of the Niagara Frontier Section, who presented the certificates, and Joe Thornberg.

Corrosion Losses Not Inevitable, F. L. LaQue Says

The magnitude of industrial losses due to corrosion may be the reason they are looked on in much the same way as are taxes, F. L. LaQue, The International Nickel Co., Inc., New York told the NACE corporate members present at a luncheon in Buffalo. The luncheon was held Tuesday, 14 March. Mr. LaQue, a vice-president of his company, formerly president of NACE and active for many years in corrosion control in NACE and other organizations, deplored the resigned attitude common in industry.

If industry would compare its corrosion losses to its profits, it would be discovered that in some years, the profits were the smaller, he pointed out. Mr. LaQue urged management to think constructively about corrosion control, and to adopt the attitude that something can be done about it.

Goodnight Names Two Men As Kansas City Chairmen

Robert H. Goodnight, Cook Paint & Varnish Co., North Kansas City, general conference chairman for the NACE 18th Annual Conference at Kansas City next March has named John E. Wright, Gas Service Co., Kansas City, chairman of the local arrangements committee and Frederic H. Rogers, Mission, Kans., chairman of the publicity committee for the conference.

Proceedings
Short Course
on
Process Industry
Corrosion

Proceedings

SHORT COURSE
on
PROCESS INDUSTRY
CORROSION

Twenty-two papers presented Sept. 12-16 at the symposium sponsored jointly by Ohio State University and NACE Technical Group Committee T-5 on Corrosion Problems in the Process Industries. The 450-page book measures 8½ x 11 inches, has a plastic back, flat opening. Many tables, graphs and illustrations; hundreds of references.

Add 65¢ per package to the prices given above for Book Post Registry to all addresses outside the United States, Canada and Mexico.

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$20 NACE Members

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These papers are by engineers concerned with controlling corrosion in some of the nation's largest chemical processing plants. Considered are use of metal and plastic materials in a wide variety of environments, including most of the common acids and alkalis; special problems found in both low and high temperature processes, corrosion measurements, coatings, design and other practical information.

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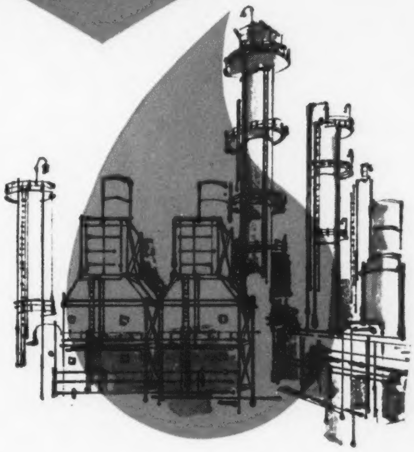
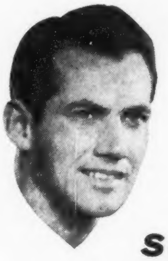


UNICOR is a potent, film-forming, oil-soluble corrosion inhibitor that plates out on metal and protects your petroleum product from contamination and your equipment from corrosion.

Whether in pipelines, refinery vessels or tanks, UNICOR provides a sure and economical means of saving dollars through assured product quality and longer equipment life. Add only 10 to 20 parts of UNICOR per million of product for lasting protection against corrosion.

With UNICOR you will eliminate contamination, get longer equipment life, reduce downtime for maintenance and cleaning, and lower power costs. For information and samples write our Products Department.



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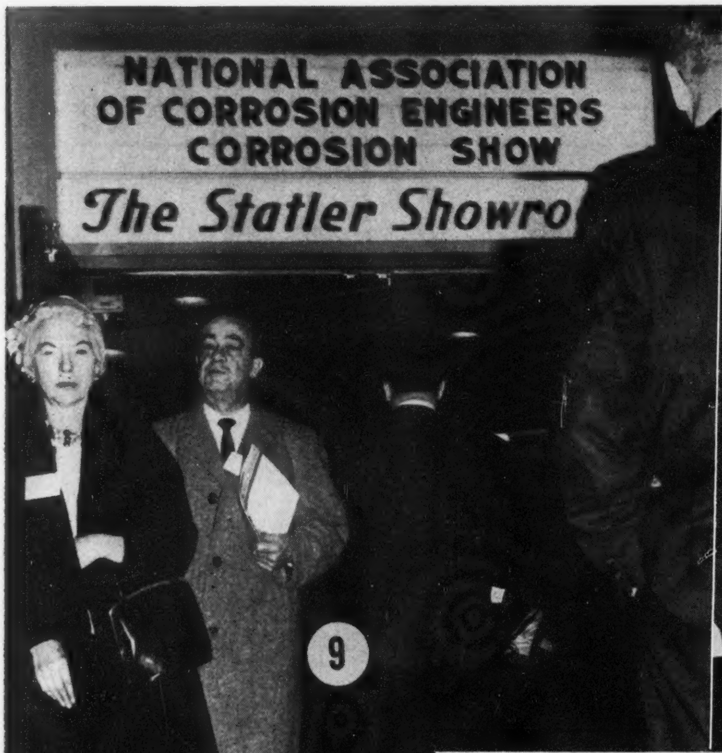

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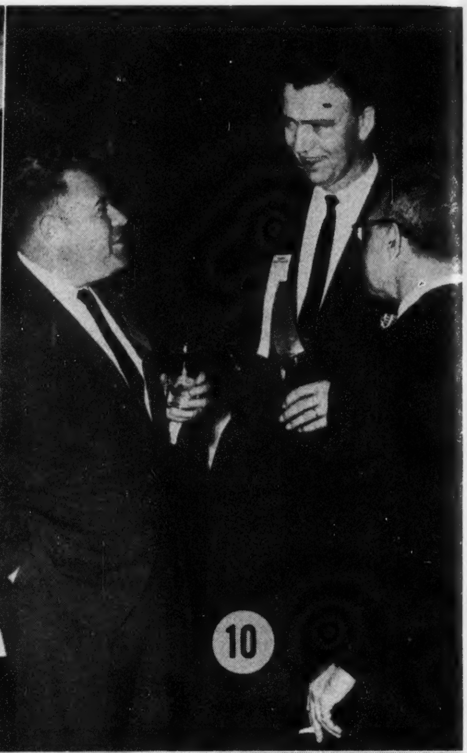

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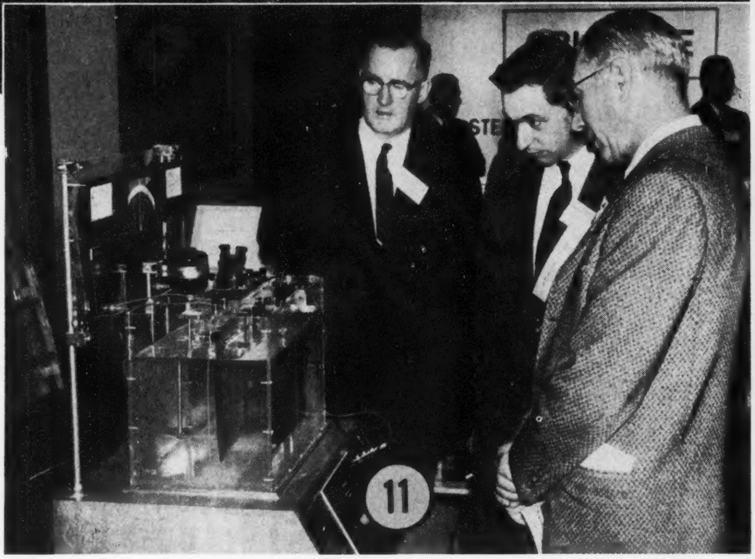

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1961 Corrosion Show

The 1961 Corrosion Show attracted a larger than usual number of visitors from the environs of Buffalo, an examination of attendance records shows. This reflects the excellent work of the conference publicity committee in securing advance publicity in newspapers and local magazines and good newspaper coverage during the show.

Many exhibitors had new booths and there were numbers of new products on display.

The 1961 Corrosion Show Slide Program is now in preparation and will be available for use in the near future. The program will consist of 60 to 65 color slides of products on display.

Photograph 9 shows the main entrance to the 1961 Corrosion Show at the Statler Hilton Hotel. Considerable local interest was shown in the 101 booths at the Corrosion Show. Photograph 10: Dick Tefankjian of Texas Eastern Transmission Corp., Shreveport, La., (at left) is talking with E. K. Benson of New York Telephone Co., Buffalo, N.Y. (in center) who was general conference co-chairman, and Sando Biondolillo of Bell Aircraft Corp., Buffalo N.Y. Photograph 11 is one of the display booths at the Corrosion Show in which a demonstration was given. This booth showed a miniature anode system in a salt water exposure, simulating the use of anodes to protect ship hulls and other metals exposed to sea water.

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Approx. Size

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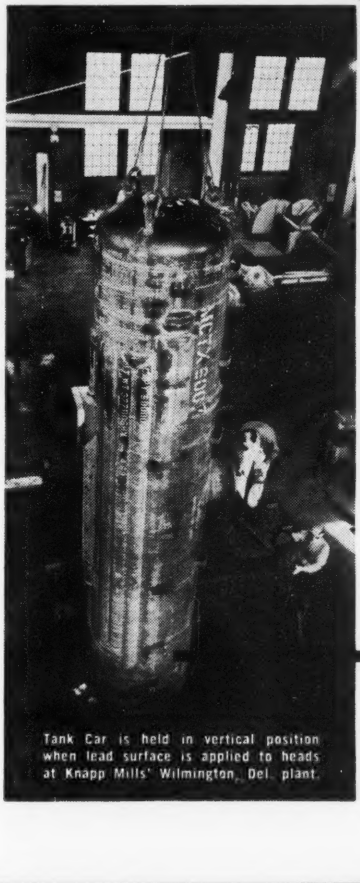
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15

Disbanding of ISCC Approved at Buffalo

The Inter Society Corrosion Committee, consisting of representatives of about 35 organizations active or interested in corrosion control was disbanded at Buffalo. The decision was reached after considerable discussion during the NACE Conference there March 13-17, when the concensus seemed to be that it no longer was effectively performing the functions for which it was organized.

Some of the activities of the society will be transferred to other committees in the association. Exactly what will be done in this connection will be announced later.

●

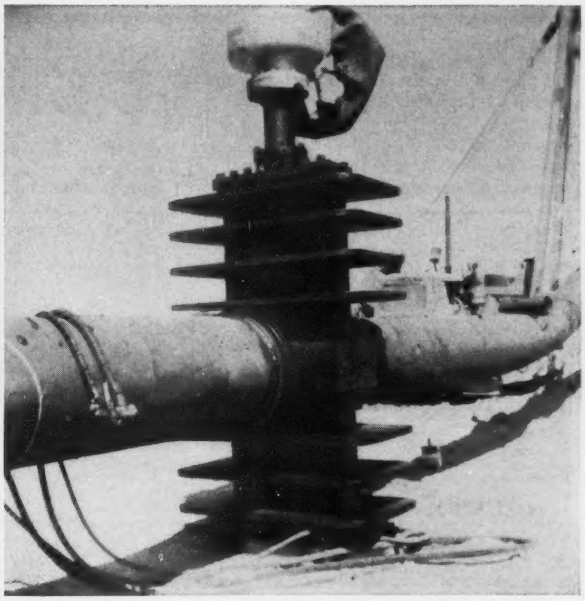
NACE's 18th Annual Conference and 1962 Corrosion Show will be held March 18-22, 1962, at the Municipal Auditorium in Kansas City, Mo.

Conference Snapshots

Photograph 12 shows one of the prolonged discussions that followed some of the technical committee meetings held during the conference. After the meeting of T-6B-17 (Chemical Cured Coal Tar Coatings), these four gentlemen continued the discussion. They are from left to right Lloyd B. Nelson of Shell Pipe Line Co., Houston, Texas, P. B. Thomas of Asiatic Petroleum Co., New York, N.Y., William Strunk of Food, Machinery and Chemical Corp., New York, N.Y., and H. C. Owens of Coast Paint and Lacquer Co., Houston, Texas. Photograph 13: Barbershop harmony courtesy of the "Corrosive Four" was not on the technical program, but these four gentlemen added something to the entertainment: the first two on the left are members of the NACE Board of Directors (E. L. Simons of General Electric, Schenectady, N.Y., and T. R. B. Watson of Corrosion Service Limited, Toronto, Canada), the third is NACE's immediate past president George E. Best, and the fourth singer was one of the local arrangements committee chairmen. Photograph 14: Another informal discussion involving (from left to right) Paul C. Hoy of Dayton Power & Light Co., Dayton, Ohio, Richard L. Seifert of Tennessee Gas Pipeline Co., Hamburg, N.Y., Carlton D. Martin of Peoples Natural Gas Co., Pittsburgh, Pa.,and Robert L. Shannon of Peoples Natural Gas Co., Apollo, Pa. Photograph 15: Herbert H. Lawson of Armco Research Center, Middleton, Ohio, and James H. Myers of the U. S. Air Force, Dayton, Ohio, conduct another informal discussion.

EL PASO NATURAL GAS COMPANY PROTECTS LINE VALVES WITH ROSKOTE COLD-APPLIED MASTIC

Roskote cold-applied mastic was spray-applied to 30" valves by El Paso Natural's personnel at Grants, New Mexico. Risers and support legs were coated with Roskote and wrapped with Royston Glas-Wrap for additional mechanical abrasion protection.



On the El Paso Natural Gas Company line being laid at Fort Stockton, Texas, by Western Pipeline Company, all 20" valves were spray-coated with Roskote cold-applied mastic.

You get superior protection against the most severe field corrosive agents with Roskote cold-applied mastic. Valves, risers, line pipe, tanks and other structures above or below ground are coated with Roskote by spray or brush without heat, flame or toxic and irritating effect on workers. The quick-drying feature of these mastics permits early backfilling. Roskote's time-saving, cost-saving application complements its low first cost to provide you with the most economical means of long-life metal protection.

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Canadian Region

Montreal Section heard J. H. Ewing of Knapp Mills, Long Island City, N. Y., speak on the uses of lead-bonded steel and Insmetal in corrosion resistant equipment at the February 14 meeting. Members of the local ASM were invited to attend the meeting.

•

The 6th Annual Appalachian Underground Corrosion Short Course will be held June 6-8 at the University of West Virginia, Morgantown.

•

Greater Boston Section will hold a corrosion short course June 20-21 at the Wentworth Institute, Boston, Mass.

South Central Region

Tulsa Section will not hold its annual Corrosion Short Course for Pipeliners this year but has it scheduled for February, 1962.

Yale W. Titterington of Corrosion Services, Inc., spoke on cathodic protection of well casing at the January 23 meeting.

•

The 1961 South Central Region Conference and Exhibition will be held October 24-27 at the Shamrock-Hilton Hotel, Houston, Texas.

•

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CSI engineers—pioneers in cathodic protection—have also developed a wide variety of special sizes and shapes to make Galvomag even more efficient. For example, the use of these special sizes in "hot spot" protection of pipe lines makes it possible to lower the number of anodes required by one-third—without decreasing the amount of protection or shortening the life of the installation.

It will pay you to check with CSI—for expert engineering and installation services, plus a complete line of cathodic protection supplies for both rectifier and anode installations. Prices are competitive. Call or write today.

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SECTION CALENDAR

April

5 Schenectady-Albany-Troy Section: Ladies' Night. Corrosion in the Home, by E. A. Tice, International Nickel Co., Inc.
10 Eastern Wisconsin Section: Latest developments in Metal Spraying Plus Plasma Arc Process, by C. J. O'Boyle of Chicago.
10 North Texas Section.
10 Central Oklahoma Section. Corrosive Properties of Clear Completion Fluids, by Charles Hudgins.
10 Kansas City Section.
10 Greater St. Louis Section.
11 Houston Section.
11 Permian Basin Section.
11 Twin City Section.
12 Tidewater Section.
12 Greater Boston Section. Corrosion in Boilers and Condensate Lines, by Douglas E. Noll.
14 San Francisco Bay Area Section. Experiences with Underground Cathodic Protection, by Kent Lundergan of Electro-Rust Proofing.
18 Cleveland Section.
18 Chicago Section. Corrosion Problems in Nuclear Reactors, by Sherman Greenberg, Argonne National Laboratories.
18 Baltimore-Washington Section. Characteristics of Hot Dip Galvanized Coatings, by H. R. Breslau.
20 Vancouver Section.
20 Alamo Section.
24 Tulsa Section.
25 Panhandle Section.
25 Southwestern Ohio Section. Steels That Fight Corrosion, by John J. Halbig, Armco Steel Corp. To be held at Connersville, Ind.

May

8 Kansas City Section.
8 Greater St. Louis Section.
8 Central Oklahoma Section.
8 North Texas Section.
8 Eastern Wisconsin Section. Corrosion Resistance of Copper Alloys and Aluminum Alloys, by Wilson Lynes of Revere Copper and Brass, Rome, N. Y.
9 Twin City Section.
9 Permian Basin Section.
9 Houston Section.
9 San Francisco Bay Area Section.
12 Rocky Mountain Section.
16 Cleveland Section.
16 Chicago Section. New Coating Resins to Combat Corrosion.
18 Vancouver Section.
18 Alamo Section.
19 Kanawha Valley Section. Roundtable Discussion led by George C. Cox and A. L. Hamstead. To be at Charleston.
22 Tulsa Section.
24 Wilmington Section. Guest speaker W. Z. Friend of International Nickel Co., Inc.
30 Panhandle Section.
30 Southwestern Ohio Section. Guest speaker J. S. Long of University of Louisville. To be held at Cincinnati, Ohio.
31 Philadelphia Section.

NATIONAL and REGIONAL MEETINGS and SHORT COURSES

1961

Oct. 4-6—Western Region Conference, Benson Hotel, Portland, Oregon.

Oct. 9-11—North Central Region Conference, St. Louis, Chase Park Plaza Hotel.

Oct. 24-27—South Central Region Conference and Exhibition, Shamrock-Hilton Hotel, Houston.

Oct. 30-Nov. 2—Northeast Region Conference, New York City, Hotel Statler.

Nov. 27-Dec. 1—Southeast Region Conference and Florida General Conference Short Course, Key Biscayne Hotel, Miami, Fla.

1962

March 19-23—18th Annual Conference and 1962 Corrosion Show. Kansas City, Missouri, Municipal Auditorium.

October 1-4—Northeast Region Conference, Hotel Sheraton Ten Eyck, Albany, N. Y.

October 9-11—North Central Region Conference.

October 11-12—Southeast Region Conference, Birmingham, Ala.

October 16-19—South Central Region Conference and Exhibition, Granada Hotel, San Antonio.

1963

March 18-22—19th Annual Conference and 1963 Corrosion Show. Convention Hall, Atlantic City, N. J.

October 14-17—South Central Region Conference, Oklahoma City, Okla.

SHORT COURSES

1961

June 5-9—Corrosion Short Course, cosponsored by NACE Greater St. Louis Section and Washington University, to be held at Washington University, St. Louis.

June 6-8—6th Annual Appalachian Underground Corrosion Short Course, University of West Virginia, Morgantown.

June 7-8—Teche Section Short Course, University of Southwestern Louisiana, Lafayette, La.

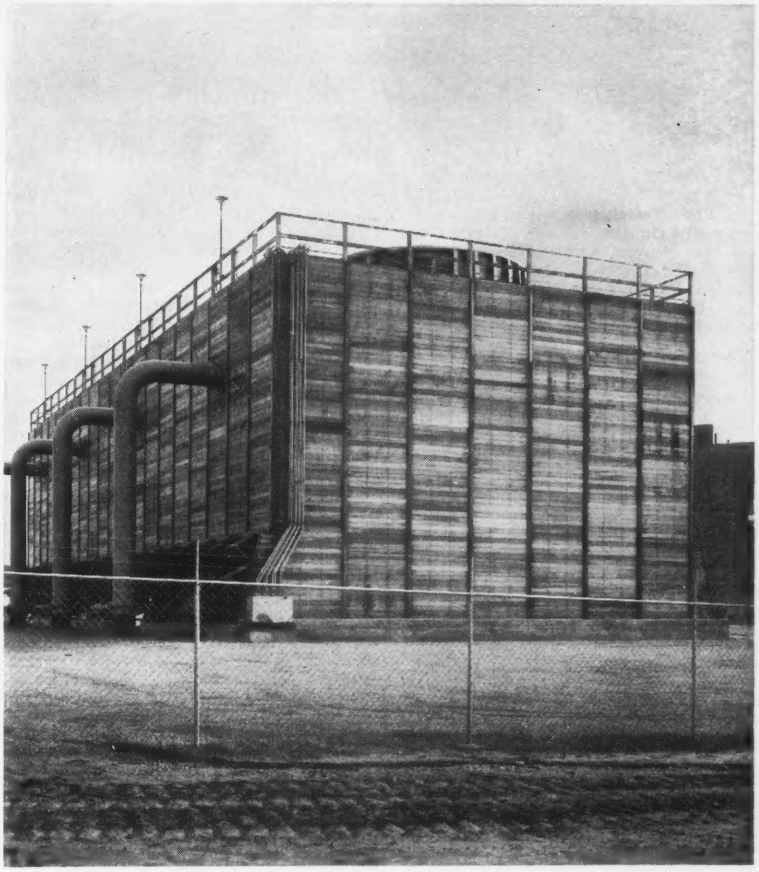
June 20-21—Greater Boston Section Corrosion Short Course, Wentworth Institute, Boston, Mass.

Sept. 27-29—8th Annual Central Oklahoma and University of Oklahoma Corrosion Control Short Course, Norman.

Fincher to Present Paper at Gas Symposium Meeting

NACE member D. R. Fincher of Tidewater Oil Co., Houston, Texas, will present a paper titled "Corrosion in Gas Wells and Gas Gathering Systems" at the AIME Society of Petroleum Engineers Gas Technology Symposium scheduled for April 20-21 in Tyler, Texas. Mr. Fincher is chairman of NACE Technical Committee T-1G on Protective Coatings and Non-Metallic Goods for Oil Field Use.

CORROSION COSTS CAN BE LOWERED



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Foreign Corrosion Reports

Four New Foreign Correspondents

Four new foreign correspondents have been named, according to Hugh P. Godard, chairman of the ISCC Subcommittee on Foreign Relations. They are to report on corrosion control activities from Spain, Norway, Austria and Sweden.

They are as follows: J. M. Alameda, Division de Electronica Especial, Marconi Espanola, S.A., Alcala 45, Madrid 14, Spain; H. Grubitsch, Technical University, Lessingstrasse 25, Graz, Austria; Tor Hurlen, Central Institute for Industrial Research, Forskningsveien 1, Blindern, Norway; and Ake Bresle, IVA:s Korrosionsnamnd, Box 5073, Stockholm 5, Sweden.

Corrosion Activity in Spain, 1960

The following report is given by J. M. Alameda of Madrid, Spain:

Good weather and low industrial development were mainly responsible for apathy toward corrosion in Spain until the last 20 years, with the exception of interest in limited areas of industrial development along the coast, such as Bilbao, Gijon and Barcelona, where the typical corrosion problems due to sulphur polluted air and salt brine were found.

The mild conditions required only decorative finishes. Very thin electroplated coatings, mildly protective paints and varnishes and an academic interest in corrosion were the rule. Only in recent years has the gradual industrial growth in petrochemical plants, automobile plants, military installations, electronics and heavy chemicals required better protective finishes and a serious interest in corrosion prevention.

With the recent industrial development and a better understanding of the implications of corrosion in both production and sales, there is a rising demand for protective products. This is being satisfied by a number of newly established firms, generally licensed or branches of foreign companies such as Parker, Walter, Valentine, Sherwin Williams, International Paints, Udylite, etc. Of course the cost of the new methods, the scarcity of foreign currency and the difficulty of securing import licenses have slowed this activity, but the situation is now improving. A major development has been the production of synthetic resin finishes which were practically unknown in Spain a half dozen years ago.

Much remains to be done in modernizing electroplating practice, but progress is slowed by an uncertainty of supply of brighteners and special chemicals.

No institution in Spain is devoted exclusively to corrosion research, although there are corrosion sections or departments in some organizations. The Instituto Espanol de Oceanografia and the Metallurgical Laboratory of Madrid University were leaders in the corrosion field and produced scientists and engineers with a knowledge of corrosion. A mainstay was Professor E. Jimeno who retired last year, leaving a vacancy that will be difficult to fill.

Of more recent origin are the following laboratories (a) Department of Corrosion, Instituto del Hierro y del Acero, Ciudad Universitaria, under Dr. Rubio. Here the major subjects of investigation are electroplating and conversion coatings for iron alloys. (b) Laboratory of Electrochemistry, Instituto de Fisico-Quimica Rocasolano, Madrid, under Professor Ruis, with a program of basic research in electrochemistry, some connected with corrosion. (c) Department of Corrosion and Electrochemistry, Instituto de Metales no-ferreos, Madrid, under Dr. Serra, especially devoted to cathodic protection.

In 1956 the Asociacion Nacional de Quimicos de Espana formed a Seccion de Corrosion y Prevencion de Materiales to coordinate the growing interest in corrosion and its prevention. The section now has about a hundred members distributed in branches at Madrid and Barcelona and in branches now being formed at Bilbao, Gijon and Puertollano. Activities of the section include bimonthly colloquia and conferences. An interesting recent meeting dealt with the Kanigen electroless plating process, with the talk being given by the Spanish licensee of this American process. The section maintains a consulting office and library.

In 1960 the most important activity of the section was organization of the first Corrosion Symposium to be held in Spain. This was in Barcelona October 26-27, in cooperation with the 23rd International Congress of Industrial Chemistry, and the 11th Journee de Corrosion of the European Federation of Corrosion. (See CORROSION, January, 1960, Page 43.)

The section will welcome enquiries from abroad and will provide information and corrosion contacts in Spain upon request. These should be addressed to Seccion de Corrosion, Asociacion Nacional de Quimicos de Espana, Calle de Lagasca 81, Madrid 6, Spain.

PDC Activities 1960

C. J. Wessel, Director
Prevention of Deterioration Center

The Prevention of Deterioration Center, a project of the Division of Chemistry and Chemical Technology of the National Academy of Sciences-National Research Council, acts as an information, advisory, consulting, publications, liaison, and conference host center in the field of prevention of deterioration of materials and equipment used by the Department of Defense. The work is sponsored and financed by the Air Force, Army and Navy through a contract between the Office of Naval Research and the NAS-NRC.

Among the materials and deterioration phenomena in its scope are metals and corrosion. These are served chiefly by (1) Publications. (2) Question answering or information service. (3) Corrosion Subcommittee, and (4) miscellaneous.

During 1960 activities were as follows:

1. **Publications.**

a. The Prevention of deterioration Abstracts, Vol. XVII, Issues 7-12, and Vol. XVIII, Issues 1-6, contained 284 abstracts and 160 extracts on corrosion of metals, totaling 444.

b. The PDC Newsletter contained listings of about 70 recent corrision articles of interest. Also included were a feature article with a bearing on the corrosion field, notices of corrosion meetings, courses, announcements of corrosion textbooks, notes on miscellaneous corrosion items and news items of general interest to corrosion engineers.

2. **Question Answering or Information Service.**

More than 30 separate inquiries on corrosion control subjects were answered.

In addition to these more or less specific requests there were numerous requests for information associated with the field of corrosion, such as protective coatings for metals, marine fouling problems, organisms associated with bacterial corrosion, sulfate-reducing bacteria, wood preservation studies, etc. Numerous requests were made for loan of published articles on corrosion subjects or subjects indirectly related to corrosion from our literature files.

3. **Corrosion Subcommittee.**

During 1960 the composition of the PDC Corrosion Subcommittee was altered. Herbert H. Uhlig, former chairman resigned his post because of his leave of absence from MIT for a sabbatical year in Europe and Frank L. LaQue, The International Nickel Company became chairman. New members are Morris Cohen, National Research Council of Canada, and George Best, Manufacturing Chemists Association.

There were no occasions this year to call upon the services of the Corrosion Subcommittee. However, one member, Mr. Best, was approached for possible support by the NACE of a publication effort by the American Institute of Biological Sciences to publish a general bibliography on marine boring organisms. This matter was brought to the attention of Mr. Hull of the NACE and a decision was arrived at by NACE, transmitted by Mr. Best to PDC and from thence to the AIBS. The decision was that NACE was unable to support this publication effort.

4. **Miscellaneous.**

Staff members attended and participated in several meetings concerned in part with corrosion control.

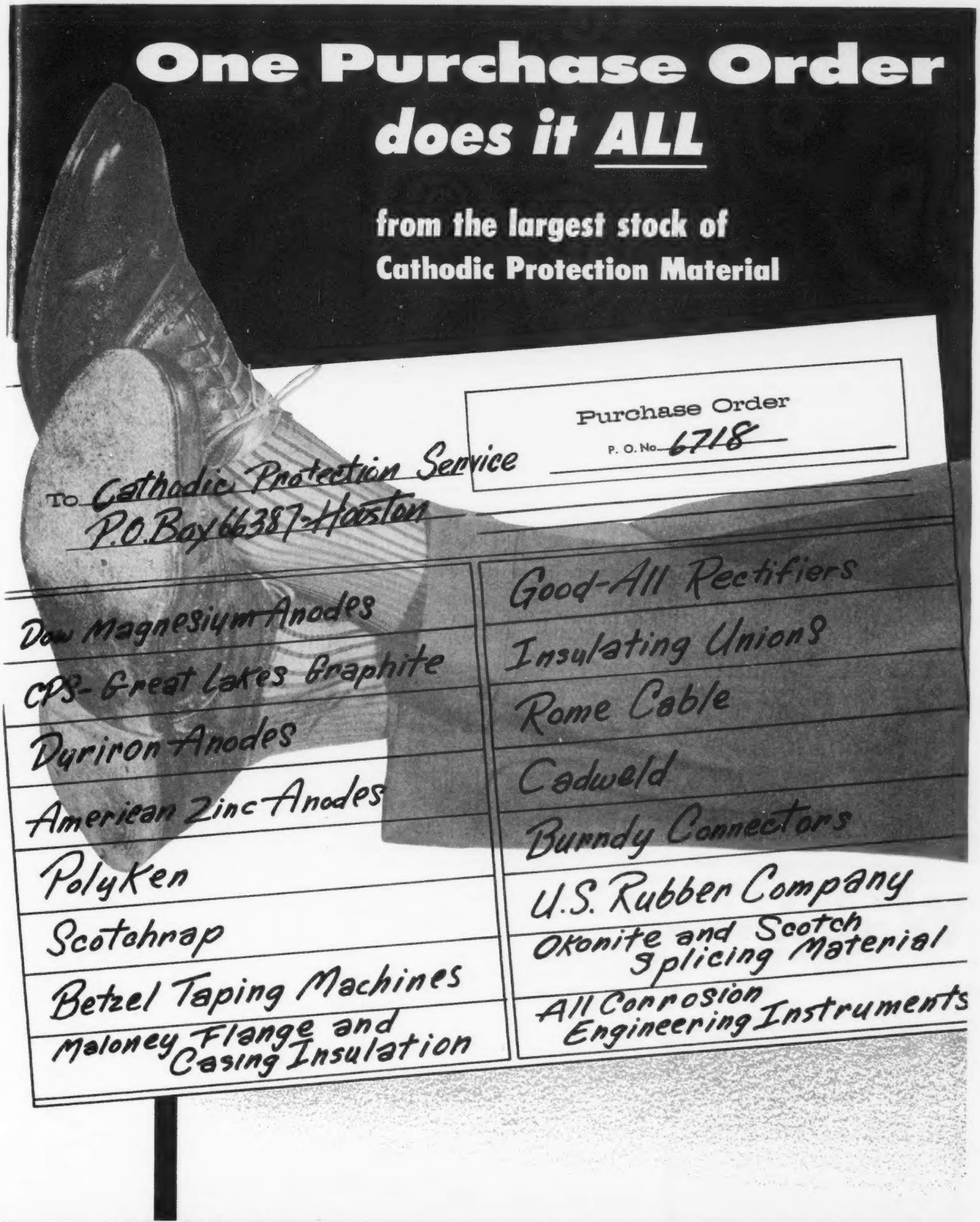
The Center has continued to provide NACE with copies of the PD Abstracts regularly. We also provide copies of the PDC Newsletter to the NACE office.

PDC has been given responsibility in the field of the "induced", "hyper", or "space-associated" environments. In June 1960, the funds of the Center were increased to permit entrance into this field. The environmental factors included in these environments are shock and vibration, extremes of temperature, dissociated and ionized gases, vacuum, meteoritic dust, electromagnetic radiation, conoras, auroras, and associated factors.

First step to be taken by PDC in servicing this field is publication of new abstracts series, "Environmental effects on Materials and Equipment". Volume 1, Issue 1, 36 pp, was published and distributed on January 15, 1961.

Although subject matter of this publication is **not directly** in the field of corrosion, many topics discussed are closely associated with corrosion and other environmental deterioration of materials and equipment. The publica-

(Continued on Page 70)



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Letters to the Editor

Magnesium Elektron Limited
Swinton, Manchester

The Editor, CORROSION
NACE
Houston, Texas, U.S.A.

I would like to comment on Mr. Keynes letter in your issue of December, 1960.

Mr. Keynes is an economist and is no doubt entitled to ask for consideration of all forms of wastage, but his opinions on corrosion as a form of wastage need to be examined carefully. There is a naive opinion abroad in the world that corrosion can be prevented. It cannot,—it can only be avoided, and the distinction is extremely important. It is into this error that Mr. Keynes appears to have fallen. The fact is that the corrosion specialist may study corrosion and may even understand it, but he cannot really cure it,—he can only take evasive action and this may be just as expensive or even more expensive than the corrosion itself. There are of course exceptions, but when you examine the matter closely I think you will agree that what I say is basically true.

I believe there will be many objections to this statement, however, and even the most patient of your readers will point to cathodic protection, galvanizing and painting and so on as evidence of what can be done to prevent corrosion. I concede the point—up to a point—but have we really prevented corrosion by these means? In two of the cases, only by life-long vigilance, by maintenance, and in the third case we have merely exposed a more enduring metal to the condition—for a while. In one of the cases indeed we have merely offered an exchange.

Corrosion is a slow eating away of a metal by the environmental influences. The only way to *prevent* it is to change the conditions or substitute a more resistant metal. In the great bulk of industrial processes both measures are usually impracticable. The "conditions" are usually part and parcel of the process and cannot be modified to any worthwhile extent. If you produce a corrosive hygroscopic dust in a chemical process, you are likely to get corrosion of the structural steel. It may be said that you can confine the dust or dry the air or substitute something else for the steel, but this is rarely an economic solution.

Thus the problems of corrosion all boil down to the same basic considerations; which is more economical in the long run—to build with commonly available materials cheaply and continue to repair or replace, or to build with rarer and more costly metals and be free (or at least less harassed) from problems of maintenance? The answer must be decided individually in almost every case and judicious compromise is all that the corrosion specialist can usually achieve for his firm. There are other factors, too, Mr. Keynes letter suggests that if all corrosion could be prevented or controlled a great financial saving could be effected. This is not necessarily true. The figures for corrosion costs quoted from various learned sources may or may not be true, but the converse is not true and these figures would certainly not represent the *saving* even if all corrosion could be entirely prevented. What of obsolescence? Very few manufacturers are far sighted enough to scrap a plant and build a new one even when they know their existing plant is out of date and uneconomic. Corrosion sometimes forces their hand to their own ultimate profit. And what of the automobile industry? Would prevention of corrosion of motor cars, and especially their bodies, help or hinder? It is a debatable point but the fashionable change to the new season's model is often due to the shabbiness (corroded condition) rather than to any intrinsic virtues in the new model.

One final point—corroded metal is not entirely lost. Corroded plant, fit only for the scrap heap, is still the richest form of ore and even if 50% of the original metal has vanished as loose rust, a condition which must be rare indeed, the scrap metal compared with raw ore is extremely valuable.

No, Mr. Keynes. Let us economise in our natural resources by all the means we can, but let us keep the problem in perspective. Corrosion research is of vital importance in controlling and directing the proper choice of materials, the proper design of equipment, and the proper operation of plant. This is especially true in those industries where the value of the operation is very large compared to the value of metal content of the plant. But corrosion research cannot be turned into an economic tool which would materially conserve the steel needed for the building of bridges or large factories or the like. In expecting a return from corrosion prevention Mr. Keynes is probably playing with a small fraction of a percent.

Yours faithfully,
W. F. Higgins

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Foreign Corrosion—

(Continued From Page 68)

tion is being offered free of charge to military offices and personnel and is available to others at $25.00 per year.

The Center has broadened its concepts considerably on just what deterioration is and what the causes of it might be. This is reflected in the added responsibility described above. If deterioration as caused by the "natural" and the "induced" environments is combined, it becomes quite evident that the tempo of work on deterioration prevention has increased very greatly in magnitude. The environment, and the deterioration it causes, are being studied as never before. This includes some fairly fundamental research, much applied research, and a great deal of specification and other testing, especially in the field of missiles, rockets and satelites. Much interest is being evidenced in combing the literature available both at home and abroad including the Iron Curtain and Communist China journals.

The high pitch of interest is evidenced in a document put out by the Office of Fuels, Materials, and Ordnance, Office of the Director Defense Research and Engineering, entitled "Summary of Recommendations for Research and Development in Materials", July 1960. Here much emphasis is placed on research and development in the deterioration of materials and structures, engineering guidance and guidance studies, and such specific things as the need for more and different research in corrosion phenomena, high temperature materials, erosion of metals and other materials, and the development of improved test equipment and techniques.

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CORROSION ABSTRACTS

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4. CORROSIVE ENVIRONMENTS

4.3 Chemicals, Inorganic

4.3.3, 6.2.3, 3.4.8, 8.5.3

Corrosion of Mild Steel in Alkaline Pulping Liquors. Pt. 3. Special Effect of Sulfate Ion in Kraft White Liquor. R. B. Kesler. Inst. Paper Chemistry. Paper before TAPPI, 14th Eng. Conf., Pittsburgh, Oct. 12-15, 1959. TAPPI, **43**, No. 4, 355-357 (1960) April.

In earlier work it was observed that dual corrosion rates of mild steel in kraft white liquor were obtained at constant liquor corrosivities, when corrosivity values were calculated according to Roald's corrosivity equation. Prediction was made that a family of curves would be obtained by plotting liquor corrosivity against corrosion rate, each curve being specific for a definite sulfite ion concentration. Results are presented of experiments that substantiate prediction; and further define role of sulfite ion in steel corrosion in white liquor. A six- or sevenfold increase in corrosion rate occurred upon a reduction in Na sulfite, in liquor, of only 0.3 g.p.l. Linear relationship between liquor corrosivity and steel corrosion rate begins to break down as Na sulfite content of liquor is reduced to about 1.5 g.p.l. Graphs, table, 6 references.—INCO. 20057

4.3.5, 4.2.3, 3.2.2

Effect of Environment on High Temperature Creep Properties of Metals and Alloys. Donald A. Douglas. (Am. Inst. Mining and Metallurgical Engrs. Conf. on) High-Temperature Materials, 1957, **1959**, 429-447.

Effects of environments leading to oxidation, reduction, corrosion and nitriding are discussed. Premature failures in gas-turbine engines are often due to accelerated oxidation, promoted by vanadium or lead in the fuel. Structural failures due to corrosion can often be traced to the presence of reducing agents, e.g., hydrogen or H_2S. An atmosphere containing nitrogen can lead to increased creep strength if nitrides are formed, e.g., in Inconel. 23 references.—MA. 20172

4.3.2, 6.2.5, 6.3.10

Corrosion of Stainless Steels in Fuming Nitric Acid. (In French.) Michel L. J. Bernard. Corrosion et Anti-Corrosion, **7**, 373-383 (1959) Nov.

Nineteen martensitic, ferritic or austenitic stainless steels and three nickel alloys were tested in concentrated nitric acid at ordinary temperature and in boiling nitric acid at 86 C. Influence of composition of acid, nature of metal, time, temperature and pressure. Only better grades, such as 18-10 types, with low carbon content, are sufficiently resistant in boiling acid. 11 references.—RML. 20228

4.3.3, 4.2.3, 2.3.4

Corrosion by Vanadium-Pentoxide Sodium Sulphate Mixtures in Laboratory Tests: Correlation With Corrosion by Oil-Ash in Field Tests. H. Lewis. British Petroleum Equipment News, **7**, 48-55 (1959-1960) Winter.

Laboratory production of corrosion similar to that caused by ash deposits in oil-fired boilers as a means of determining which substances are responsible for corrosive effects.—RNL. 19779

4.3.3, 6.2.3, 6.2.4, 6.2.5, 8.5.3

Corrosion Due to Black Liquor and Its Vapors. L. Stockman and A. Tansen. Svensk Papperstidn., **62**, 907-914 (1959). (In Swedish with German and English Summaries.) Abstr. Bull. Inst. Paper Chem., **30**, 1164-1165 (1960); Paper Ind., **42**, No. 3, 215 (1960) June.

Corrosion of five different grades of steel used in evaporator tubes—"killed" and "rimmed" C steel, 5 Ni steel, 18-8, and 13 Cr steel. Rate of vapor phase corrosion was studied both in case of oxidized and unoxidized black liquors, and in vapor phase in which 1% oxygen had been added. Corrosion was studied as function of sodium sulfide content of unoxidized liquor. 5 nickel steel was attacked far less in vapor phase than were carbon steels, but in liquid phase it showed its superiority only when sulfide concentrations were quite high. 13 Cr. and 18-8 were very resistant to attack in both phases. There was a marked decrease in vapor-phase corrosion by addition of a film-forming amine inhibitor.—INCO. 19986

4.3.7, 3.7.2, 3.4.8, 8.4.3

High-Temperature Corrosion of Oil Refining Equipment by Sulfur Compounds. (In German.) W. G. Richards. Werkstoffe u. Korrosion, **10**, 74-77 (1959) Feb.

Corrosion of alloy steel (8% chromium content) in hydrogen sulfide at 780 F amount to 0.13 in. per year. Increase in chromium to 30% decreases speed of corrosion to 0.01 in. per year.—RML. 18518

4.6 Water and Steam

4.6.1

Corrosion by Hot (Saline Spa) Waters. Enrico Chiostri. Corrosion et Anti-Corrosion, **8**, No. 1, 15-36 (1960).

Presented at a meeting of the Association Nationale des Ingénieurs Sanitaires, Naples, June, 1957. Practical experiences, and data from tests under particularly severe conditions at up to 100 C are presented, illustrated, and discussed with reference to corrosion of the extensive feed, effluent, etc. systems at the Montecatini (Italy) medicinal baths. The aggressivity of the warm (34.4 C) strongly saline (NaCl 11.7, Na_2SO_4 2.16, $CaCl_2$2g/1), natural waters is heightened by their gas content. In the tests, cast Ni-Resist (Ni 14, Cu 6%), light alloys, Zn, Al, Mg, brass, Cu, and Fe were soon corroded, Hastelloy D and cupro-nickel less quickly so, and good resistance was shown by Ni, Pb, Hastelloy C, Monel metal and some bronzes. Pb pick-up by the waters is not an aspect from the toxicity point of view, owing to the rapid formation of a salt film on the inner walls of lead piping. Cost and other considerations of the various materials are considered.—MA. 20273

4.6.4, 7.4.1, 3.3.2, 5.8.1

How Panhandle Eastern Combated Five Common Cooling-Water Problems. J. T. Russell. Panhandle Eastern Pipe Line Co. Oil and Gas J., **58**, Nos. 5, 7, 101-102, 104-108, 116-117, 120 (1960) Feb. 1, 15.

Research work is described which it is believed provides solutions to four problems—scale, corrosion, attack of cooling-tower lumber by chemicals in cooling water, and waste disposal—as they relate to open system cooling water. Fifth problem, that of biological fouling and decay, is yet to be solved, but mention is made of work in prog-

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4.6.1, 6.4.2, 3.5.9, 3.7.2

High-Temperature Aqueous Corrosion of Aluminum-Plutonium and Aluminum-Silicon-Plutonium. H. C. Bowen. General Electric Co., Hanford Atomic Products Operation. U. S. Atomic Energy Commission Pubn., HW-61345, July 31, 1959, 8 pp. Available from Office of Technical Services, U. S. Department of Commerce, Washington, D. C.

Short-term corrosion tests were carried out on aluminum-plutonium and aluminum-silicon-plutonium alloys in 350 and 360 C water. Bare as-cast and bare extruded alloys of varying plutonium content were tested for durations of one day or less. Aluminum-plutonium as-cast alloys containing less than 6% plutonium have little resistance to rapid high-temperature attack, but the resistance increases with plutonium content up to at least 13% plutonium. Working of the material drastically decreases its resistance to attack compared with as-cast material of the same plutonium content. Addition of 12% silicon to aluminum-plutonium alloys containing up to at least 15% plutonium makes them resistant to attack for not less than 24 hours and makes them less sensitive to working.—MA. 20086

4.6.6, 4.6.7, 8.1.4

The Influence of Corrosion on the Quality of Drinking Water. L. Rott, J. Steinmetz, G. Szabó and L. Almassy. In Hungarian: Orvosi Szemle, **6,** 333-336 (1958); In Rumanian: Rev. Med., **6,** 336-338 (1958). Chem. Absts., **53,** No. 7, 6492h (1959) April 10.

Reasons were sought for turbidity, yellowish color, and unpleasant taste of drinking water supplied to town by a 1955 treatment installation. Water originates from a river, is purified by coagulation with Al sulfate, and brought into town by a 14 km steel pipeline. A neg. stability index, high concentration of CO_2, and increased iron concentration indicate corrosion processes in pipeline, due to fact that in soft river water purified with $Al_2(SO_4)_3$, CO_2 is liberated. —AWWA. 19438

4.7 Molten Metals and Fused Compounds

4.7, 6.2.3, 6.2.4, 6.2.5

Corrosion of Steels in Liquid Bismuth and Lead. J. A. James and J. Trotman. J. Iron and Steel Inst., **194,** Pt. 3, 319-323 (1960) March.

Corrosion of a variety of steels in liquid Pb and Bi was studied at 600-900 C by measuring a weight losses of steel pins after immersion in hot side of a thermal convection loop. In uninhibited Bi, low-alloy steels are more resistant to corrosion than 13 Cr-Fe or 18-8. Both Ni and Mn have an equally deleterious effect on corrosion resistance of 2¼ Cr-1 Mo steel. In Bi inhibited with Zr, best steels tested had extrapolated corrosion rates less than 0.005 in/yr at 700 C with a 50 C temperature differential. Corrosion of steels in uninhibited Pb is about 40 times less than in uninhibited Bi under similar conditions, and corrosion is reduced by addition of 500 ppm Ti, with same order of steel resistance as for inhibited Bi test. Analyses of steels tested are tabulated. Included were CRM6 steels; mild steel Cr-Mo, Mo-V, and Cr-V-W alloy steels 18-8; and 13 Cr-Fe. All steels contained Ni, with exception of one CRM6 type. Sketches of test apparatus, tables, photomicrograph of corroded corner of a CRM6 steel pin showing localized attack of pitting at corner, and 9 references.—INCO. 20117

4.7, 6.2.1

Corrosion of Iron and Steels in Liquid Metals. Pt. I Static Corrosion Test. (In Japanese.) Y. Imai and T. Ishizaki. J. Atomic Energy Soc. Japan, **2,** 96-101 (1960) Feb.

Modes of attack by liquid metal are direct alloying, dissolution, intergranular penetration, corrosion by contaminants, and others. Microscopic examination of metallographically polished surface was adopted to investigate the attack of liquid metals on iron and steels. The reliability of the experimental method, the effect of oxygen and the effect of stabilizer of oxygen were examined.—NSA. 20118

4.7, 4.2.3, 2.3.7, 2.3.9

Some Techniques of Corrosion Testing in Molten Materials. W. M. Spurgeon, C. H. Philleo and B. A. Thompson. General Electric Co. Ind. & Eng. Chem., **52,** No. 1, 109A-111A (1960) Jan.

Methods described were used in studying corrosion of aircraft gas turbine materials by condensed-phase combustion products, mainly molten boric oxide plus a little free B, from high energy fuels. These methods are general and apply to corrosion studies with other molten materials. Typical molten corrosive media, factors limiting service life of metals in molten media (rupture creep, fatigue, corrosion and combination effects), and types of tests are tabulated. Tests methods are given for specimens without and with load, with liquid stationary and liquid moving, and include static and dynamic immersion tests, corrosion-rupture, corrosion-creep, and corrosion-fatigue tests. Techniques for evaluation of results are discussed. Mention is made of usefulness of autoradiography for supplementing metallographic method of studying diffusion into metal. Autoradiograph shows extent and paths of diffusion of B into Nichrome V after six hours of exposure to corrodent (boric oxide plus free B). —INCO. 19933

4.7, 6.2.5, 8.4.5

Trial Manufacture of an Experimental NaK System. (In Japanese.) Y. Imai, K. Fukushima and T. Ishizaki. Tôhoku Univ., Sendai. J. Atomic Energy Soc. Japan, **2,** 127-135 (1960) March.

From the results of static corrosion experiments, it was found that the stainless steels of Types 304, 316, 347 and LCN155 can be used successfully as container materials for NaK. The present investigation was carried out to confirm the safe use of such materials under conditions of dynamic corrosion as well as to get information on the technical difficulty in manufacturing an experimental NaK system. A system was designed to operate up to approximately 600 C with Type 347 stainless steel as piping. Heat was supplied by electric heater and removed by air cooler. A regenerative heat exchanger was provided to reduce the required power input; the pump used was of the a-c Faraday type. In this system, NaK was circulated at the rate of about 100 g/sec, measured by d-c electromagnetic flow meter. An expansion tank and a sump tank were also provided. "Resistance-type" and "impedance-type" level indicators were used successfully. Wetting of the pipe surface was found to affect both the heat transfer of heat exchanger and the output voltage of the flowmeter. Wetting can be attributed to the reduction of oxides and precipitation of materials (oxide or others) on the pipe surface. (auth)—NSA. 19927

5. PREVENTIVE MEASURES

5.3 Metallic Coatings

5.3.4, 5.4.5

Metal Coatings as Protection Against Corrosion. R. N. Ridge. Australian Corrosion Engineering, **3,** No. 7, 19-27 (1959) July.

In a discussion of coating methods, besides hot dip, electrodeposited, clad and sprayed coatings, a section is devoted to zinc dust/silicate compositions and zinc rich paints. It is said that by incorporating finely divided zinc powder in an inorganic silicate vehicle a composition is obtained which has advantages in common with sprayed metal coatings—freedom from heat distortion, ease of reinstatement of welds or damaged areas and applicability to field work. It is also said to be generally cheaper than sprayed zinc for an equivalent metal coating weight and does not require sealing. With regard to zinc dust paints, it is mentioned that they may blister in sea water due to the production of alkali next to the steel surface. However, they provide good protection for the steel. In the section on the selection and specification of metallic coatings, prominence is given to zinc, cadmium and aluminum. It is considered that zinc is the most suitable metal coating for protection against corrosion under atmospheric or marine conditions, having better overall corrosion resistance, being versatile and relatively cheap, and being able to prevent rusting of small areas of exposed steel when in contact with an electrolyte of sufficient electrical conductivity.—ZDA. 19997

5.3.2

Corrosion-Resistance of Copper/Nickel/Chromium Systems. A. H. Du Rose and R. T. F. McManus. Electroplating, **12,** No. 11, 409-415 (1959).

A copper undercoat to a dull nickel or to a sulfur-free nickel deposit is of no practical benefit, but may greatly improve the corrosion-resistance of sulfur-containing deposits. Sulfur-containing bright or semi-bright nickel deposits are inferior to sulfur-free deposits, but a duplex of the two types perform better than comparable thicknesses of either type alone. The high-temperature (130-150 F; 55-65 C) chromium bath was found superior when heavy deposits were applied.—MA. 20073

5.3.4

Chemistry of Bright Nickel Plating Solutions. E. B. Saubestre. Enthone, Inc. Plating, **45,** No. 13, 1219-1227 (1958) Dec.

Brighteners in nickel plating solutions fall into 2 categories. Those of first class provide bright plate over bright substrate and permit second class brighteners to be present over acceptably wide range of concentrations. Brighteners of second class make it possible to build mirror-brightness, but yield brittle, stressed deposits of poor appearance, and intolerant to impurities in plating solution. These objections are overcome by adding brighteners of both classes. Chemical groupings characteristic of the two classes are tabulated. Role of leveling agents is mentioned. Importance of cathodic reduction and anodic oxidation of brighteners is stressed with many typical reactions. It is shown how sulfur may appear in nickel deposits. Reaction of brighteners, and their oxidation-reduction products, with nickel to form various nickel-organic complexes is discussed, and implications for nickel plating presented. How brighteners may affect acidity of boric acid in cathode film is explained. Principles of compounding brighteners, and their effect on properties of bright nickel deposits are summarized.—INCO. 17699

5.3.4

Effect of Pretreatment of Zinc Immersion Method on Surface Roughness of their Metal Coatings on Aluminum. (In Japanese.) Naoharu Kinoshita, Masao Watanabe and Isoji Toyoshima. J. Metal Finishing Soc., Japan, **10,** No. 2, 10-20 (1959) Feb.

Although a number of suitable methods for plating other metals on aluminum have recently been developed, few researchers have investigated plating on aluminum and its surface roughness. It is also true, today, that it is sometimes necessary to get the surface as smooth as possible, keeping up the initial surface roughness of aluminum basis, as in the case of electroforming industry.

Authors experimented plating on aluminum pretreated by means of zinc immersion method, and examined the surface roughness of basis and plated coatings at each stage of the plating process.

Result is as follows: If the suitable conditions for plating on aluminum are selected, it is possible to deposit the other metals on aluminum, where the plated coatings show the same surface roughness as that of the aluminum basis. 17789

5.4 Non-Metallic Coatings and Paints

5.4.5

Organic Finishes for Metal. Pt. IV. B. M. Letsky. Product Finishing, **12,** No. 10, 94-96 (1959).

Continues the series with references to modern organic finishing systems applicable to the metal used in domestic equipment. There is detailed reference to urea/alkyd, melamine/alkyd and styrenated alkyd (polyester) finishes.—RPI. 19786

5.4.5, 3.8.4

Filiform Corrosion of Painted Steel Plate. H. Käsche. Werkstoffe u. Korrosion, **10,** No. 11, 668-681 (1959).

Experiments to elucidate the mechanism of filiform corrosion are described. Corrosion was rapidly produced under a N. C. lacquer film by first depositing on the steel surface a uniform layer of sodium chloride granules from CCl_4 suspension. The course of corrosion was followed by microscope observations and related to atmospheric

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conditions (e.g., in an atmosphere of N_2), surface pretreatment, size of sodium chloride granules, etc. Corrosive attack commences with the formation of liquid droplets, e.g., around hygroscopic impurities on the surface, and the presence of solid corrosion products formed tends to drive the droplet about the surface, which is only superficially etched, with detachment of the paint film along the path. A sharp phase separation exists between the droplet and the solid corrosion products of the path, the droplet being green and the products red-brown. The boundary is thus a reaction zone in which ferrous ions are oxidized to ferric with production of a rapidly drying hydroxide precipitate. Consideration of oxygen partial pressure in the dry rust line, oxygen and iron concentration in the liquid in the end of the moving thread and reaction rates, leads to an explanation of the kinetics of corrosion and of the characteristic V-shape of the end. The constancy of the volume of the droplet at the end, which is the cause of the filiform structure, may be established from the study of the thermodynamics of the process.—RPI. 20082

5.4.2, 6.2.5, 3.5.9, 2.3.7

Protective Coatings for High-Temperature Metal Alloys. C. G. Harman, E. F. Fowler and L. S. O'Bannon. Battelle Memorial Inst. U. S. Atomic Energy Commission Publication, BMI-804, December 15, 1952 (Declassified Nov. 20, 1959), 23 pp. Available from Office of Technical Services, Washington, D. C.

Glasses, Li_2O, CoO, Na_2O, SrO and SiO_2, with and without additions of clay were studied as coatings for stainless steel. The coatings were subjected to tests for adherence, spalling or flaking due to thermal-shock cycling and protective properties in air at 1900 F. A glass composed of 26.9% Na_2O, 21.5% SrO, and 51.6% SiO_2 was found to have good adherence, fair resistance to spalling, and appeared to have fair protective properties. (auth)—NSA. 20111

5.6 Packaging

5.6.2, 5.8.4

Temporary Coatings for Corrosion Protection. D. H. Weatherley. Corrosion Prevention and Control, **6**, No. 9, 36-40, 54 (1959) Sept.

Discusses properties and applications of spray packaging methods (Cocoon, Texikoon, Koonkote) and strippable coatings (Liquid Envelope, Spraylat, Gloscoat, Propac, Birlon). Use of vapor-phase inhibitors and inclusions of suitable desiccant (silica gel or Sorbsil) is emphasized.—INCO. 19556

5.6.2, 8.9.5

Spray Packaging and Marine Corrosion. Corrosion Prevention and Control., **4**, No. 12, 41-43 (1957) Dec.

Two main processes for the protection of equipment at sea or when a vessel is being laid up are the "Cocoon" and "Koon-Kover" processes. The Cocoon system consists of a plastic film sprayed direct by spray gun on to the equipment, while Koon-Kovers are designed as loose covers to protect an object, usually while a ship is actually at sea or in harbor.—BTR. 16166

5.6.1, 5.8.2, 8.10.4

Characteristics of Vic Materials when Applied as Packaging Media. F. H. Wayne. Rock Island Arsenal Lab., Rept. No. 59-464, Feb. 18, 1959, 15 pp. Order PB 143440 from Library of Congress, Photoduplication Service, Publications Board Project, Washington 25, D. C.

Covers results observed during the examination of Cal. .30 M1 carbines. These weapons had been in unheated warehouse storage for four years, plus a two year period in open exposure (tarpaulin covered). These carbines were packaged with four different types of volatile corrosion inhibitors used as the preservative medium. The four different types of inhibitors furnished adequate protection to the carbines and component parts during the exposure period. There was no visible evidence of rust on any of the metallic surfaces of the carbines, slings, magazines or the oilers. The gummy substance did not cause any damage to the wood stock.—GRR. 20199

5.8 Inhibitors and Passivators

5.8.1, 6.2.2, 6.4.2, 4.3.2

Effect of 8-Hydroxyquinoline on the Rate of Dissolution of Iron and Aluminum in Hydrochloric Acid. I. E. Titova. J. Phys. Chem. (Zhur. Fiz. Khim.), **33**, No. 10, 2173-2177 (1959). (In Russian.)

Effect was studied in 0.51-2.28 N-HCl and 0.0001-0.54M-8-hydroxyquinoline (I). I inhibits dissolution of iron in HCl, and the inhibiting action increases with increase concentration of acid and of I. For Al I can behave either as a promoter of solution (at low acid and I concentration) or as an inhibitor (at high acid and I concentration). These effects are caused by hydrolysis of I and the nature of the charge on the Fe and Al surfaces.—MA. 19952

5.8.2, 8.5.3

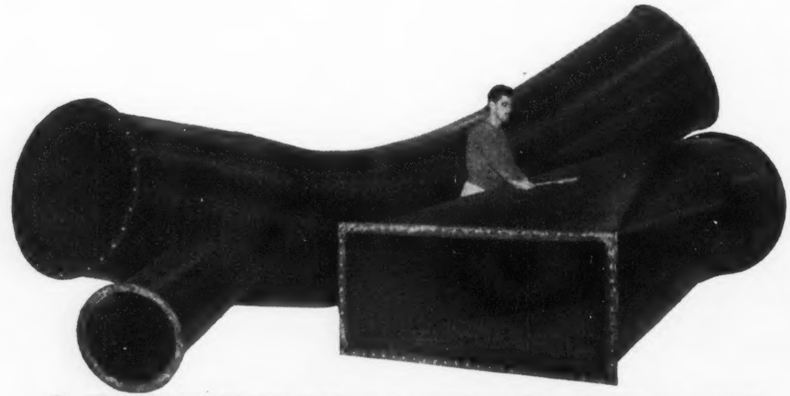
How a Corrosion Problem in a Dryer Condensate System was Solved. H. E. Carr. Hagan Chemicals & Controls, Inc. Paper Trade J., **144**, No. 12, 42-43 (1960) March 21.

Use of Hagafilm, a filming-type amine, provided answer to a tough maintenance and corrosion problem at Sumner, Washington mill of Fibreboard Paper Products Corp. Hagafilm is distilled from boiler water and transported in steam to all parts of system. From point of condensation onward, an impervious nonwettable film is deposited on all surfaces contacted by condensate rendering them immune to both O_2 and CO_2 attack. Other important benefits of filming amine are that it removes old corrosion products from lines, traps and heat-exchanger surfaces and promotes dropwise condensation. Condensate corrosion data (1952-1958) are tabulated, and results of Hagafilm treatment, as reported by Sumner mill, are given. Schematic diagram of steam condensate and raw water lines is shown. Photo shows sample steel test specimens, with and without Hagafilm.—INCO. 20133

5.8.3, 5.8.4, 6.2.3, 4.3.2

Inhibition by Organic Compounds of the Acid Dissolution of Mild Steel. Pt. 2. Distribution of Quinolines and Thioureas at the Metal/Acid Interface. J. M. West. J. Applied Chem., **10**, 250-256 (1960) June.

When the anodic and cathodic polarization curves in the absence of inhibitor are known, the individual adsorptions at anodes and cathodes may be calculated from the experimental weight losses and shifts in steady corrosion potential when inhibitor is added. An estimate is made of the effective anode



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area. Results show that thioureas adsorb generally at the interface whereas quinolines exhibit a 3:1 preference for anodic sites. 13 references.—RML. 20222

5.8.4, 7.7, 6.3.8

Studies on Chemical Corrosion of Lead-Covered Telegraphic Cables. (In Japanese.) Shizuo Nakatani, Takashi Saji and Masao Mukai. J. Metal Finishing Soc., Japan, **11,** 9-16 (1960) Jan.

Effectiveness of ammonium sulphate, sodium sulphate, sodium silicate, sodium hexametaphosphate, alpha picoline and aliphatic amines as corrosion inhibitors. —RML. 19880

5.8.1, 4.2.1

Vapour-Phase Inhibition of Atmospheric Corrosion. S. E. Sexton. Corrosion Technology, **7,** No. 5, 147-150, 162 (1960) May.

Covers development of vapor phase inhibitors, theories on their mode of action, metals protected by vapor phase inhibitors, methods of application (powder and coated paper) and examples of articles protected with vapor phase inhibitors. These include instruments, small parts, household goods, tools and machines, pipes and drums, locomotives, aircraft components, machinery aboard laidup ships and tankers, and automotive parts.—INCO. 19974

5.8.2, 7.5.4

Sodium Silicate as a Corrosion Inhibitor in Gasholders. P. G. Clements and F. R. Jackson. Gas J., **297,** 106-107 (1959) Jan.

Sodium silicate containing 29% silica as silicon dioxide was used as the inhibitor in the water tank of a gas holder of 870,000 gal capacity. Initial charge was 30 cwt silicate, equivalent to 112 ppm silica as silicon dioxide. One refresher charge of 10 cwt silicate was added during the 13 months test. Twelve test plates of unpainted mild steel were hung in each holder, six in the tank and six in the cup.

Surface water contained only a nominal amount of silica in 1 month, with a steady rise to 75 ppm in 3 months and 80 ppm in 13 months. After 3 months, there was no significant difference in the penetration between test and control panels. Maximum penetration in the tank was 0.015 in. in the test panels and 0.017 in. in the control panels; in the cup, respective values were 0.006 and 0.010 in. At six months, there was still little difference, but at thirteen months there were very marked changes between test and control panels. The figures for maximum penetration in the tank were 0.033 in. and 0.056 in. for test and control panels, respectively; in the cup, they were 0.039 and 0.049 in., respectively. Thus, in the presence of sodium silicate, the estimated service life of an unpainted ¼ in. steel plate is 7½ years in the tank and 6½ years in the cup. Comparable figures without inhibitor are 4½ and 5 years, or an improvement of 60% and 30%, respectively. The silicate treatment also markedly reduced staining of the paint work by red water.—PDA. 20220

5.8.2, 3.5.8, 6.2.5

Sodium Silicate as a Stress Corrosion Inhibitor. Technical Report 67. M. J. McGoff. MSA Research Corp., Callery, Penn. October 16, 1959, 18 pp. Contract NObs-77023.

Sodium silicate was used in combination with insulating materials to investigate its usefulness as an inhibitor to chloride stress corrosion. The corrosion specimens were Type 304 stainless steel pipes which were stressed, insulated and thermally cycled at 200 to 650 F by periodic heating and immersion into sea water. It was observed that some of the sodium silicate treated, insulated specimens exhibited no stress corrosion after about 100 hr. of thermal cycling whereas untreated specimens corroded in approximately 48 hr. The experimentation indicates that a water soluble inhibiting agent such as sodium silicate incorporated with insulation materials offers promise as a preventive technique to chloride stress corrosion. The time of protection is indefinite since the silicate appears to become spent with prolonged cycling. (auth)—NSA. 19883

5.9 Surface Treatment

5.9.3

Increased Corrosion Protection by Flame Cleaning. (In German.) H. Ehrenberg. Schweissen und Schneiden, **12,** 239-241 (1960) May.

Before cleaning, the cold steel surface is coated with a metal-sodium solution which reacts with the cold steel by precipitating insoluble metal alloys. In subsequent flame cleaning, a very thin and hard protective layer is formed on the steel surface, which serves as an undercoat for painting operations and provides an increase in corrosion resistance.—RML. 20074

5.9.4

Conversion Coatings. Oxide Films. Lester F. Spencer. Metal Finishing, **58,** 62 + 4 pp. (1960) April.

With the exception of the phosphate coatings, black oxide films are the most widely used conversion coatings for iron and steel. The coatings produced are black, adherent ferro-ferri oxide films that have moderate corrosion resistance to outdoor weathering and salt spray. Discusses coatings for copper-base, aluminum-base, and titanium-base alloys.—BTR. 20160

5.9.4

Conversion Coatings: Chromate Films. Lester F. Spencer. Metal Finishing, **58,** No. 1, 58-65 (1960).

Characteristics and uses of chromate conversion coatings and their application to zinc, cadmium, copper, brass, aluminum magnesium and silver are discussed. Chromate films give corrosion protection similar to phosphate coatings under paint. Chromate films are softer than phosphate coatings, but are self-sealing.—MA. 20280

5.9.4, 6.4.2

Hard Anodizing of Aluminium and Alloys. (In French and German.) R. Merz. Aluminium Suisse, **10,** No. 1, 21-26 (1960).

Describes the Alcoa process of producing hard coatings on Al, using a mixed electrolyte with high current density but low voltage. A coating of $\sim 50\,\mu$ thick is claimed, with a breakdown voltage of 2000-2500 V and a corrosion-resistance 4 x that of the normal sulfuric acid film. A plant installed in Switzerland is illustrated.—MA. 19920

5.9.4, 6.4.2

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nium. P. LeLong, R. Segond and J. Hérenguel. Rév. Aluminium, No. 272, 67-75 (1960).

Most processes for the formation of thick anodic films necessitate maintaining the electrolyte at a low temperature. Methods are developed involving the use of mixed organic acids which are capable of producing films up to several 100 μ thick in relatively short times, and at normal or even slightly elevated temperatures. The two preferred mixtures are $NaHSO_4$ + citric acid and $(COOH)_2 + HCOOH$. Notes are added on the most suitable alloys to be used, in both cast and wrought form, with the chief properties of the films produced.—MA. 19975

5.9.3, 5.3.4, 6.2.3

Influence of the Physical Metallury and Mechanical Process of the Basis Metal on Electroplating. Pt. V. Effect of Polishing on the Durability of Nickel-Plated Mild Steel. Maurice H. Jones and J. Zajdowski. Tech. Proc. Am. Electroplaters' Soc., **45,** 45-52; disc. 160-161 (1958).

A report on American Electroplaters' Society Research Project No. 14. The effect of mechanical polishing the mild-steel basis on the durability of Ni plating is discussed. Statistically controlled experiments show significant differences in corrosion-resistance at deposit thicknesses of 0.0005 and 0.001 in. and these are attributed to variations in surface finish. The sensitivity of the tests depends on the corrosive environment of which two types are investigated. Laboratory polishing techniques produce corrosion-resistant surfaces superior to those obtained commercially and fine finishes are generally superior to coarse finishes. For an equivalent surface roughness greased-belt polishing is superior to dry-belt polishing. 10 references.—MA. 19783

5.9.4, 6.3.19

Formation of Hard Films on Zinc and Zinc Alloy Surfaces by Electrolytic Processes. H. Fry. Metal Finishing J., **5,** No. 53, 181-189 (1959).

Progress towards a single-stage process for forming hard anodic zinc surfaces is described. Results are not yet considered satisfactory, mainly with regard to the appearance of coatings.—MA. 20192

5.9.4, 5.4.2

Sealing Anodic Oxide Coatings. Pt. I. S. Wernick and R. Pinner. Electroplating, **12,** No. 10, 373-377 (1959).

Different methods of sealing are discussed; hot-water and steam sealing are briefly described. Dichromate coatings, although unsuitable in decorative application, impart superior corrosion-inhibiting properties, and a mechanism is advanced for the process. Recommended for the highest corrosion-resistance is: $K_2Cr_2O_7$ 15, Na_2CO_2 4 or NaOH 3g, H_2O 1 liter, pH 6.5-7.5, temperature 90-95 C. Silicate coatings act as inhibitors to weak alkali solution.—MA. 20247

6. MATERIALS OF CONSTRUCTION

6.2 Ferrous Metals and Alloys

6.2.2, 3.5.9, 3.7.2

Certain Anomalies in the Properties of Ferro-Aluminum Alloys and Their Nature. (In Ukrainian.) P. P. Kuzmenko and P. V. Petrenko. Shevchenk Kiev State Univ. Ukrain. Fiz. Zhur., **4,** 497-503 (1959) July-Sept.

Results are presented of an experimental investigation of the dependence of the lattice parameter on the concentration and temperature, and on the oxidability of ferro-aluminum alloys with 16, 20, 25, 30, 35, 40 and 50 at .% of aluminum at 1200 C in air. It is shown that the anomalous dependence of the lattice parameter on the concentration is preserved throughout the temperature range investigated (up to 1000 C). There is, however, a considerable deformation of the curve of dependence of the parameter on concentration with change of temperature. The alloys investigated may be divided into groups by concentration with respect to the tendency to oxidation at 1200 C. The first embraces the interval from 16 to 20% aluminum, and the second from 20 to 50% aluminum. The oxidability of the second group depends slightly on the concentration and has a value of the order of 2 g/mk•hr with a 30 to 40 hr exposure in air. On passing from 20% to 16% the oxidability increases by a factor of 200. It is also shown that with a deformation of alloys with 35, 40, 50% aluminum the alloys become ferromagnetic. Subsequent annealing at 700 C for two hours leads to the disappearance of ferromagnetism. An explanation of the nature of the anaomalous properties of the alloys is given from the standpoint of superposing ionic coupling due to redistribution of valent electrons among the component atoms in the alloy. (auth).—NSA. 19996

6.2.2, 3.2.3, 3.5.9, 2.3.9, 3.7.2

Electron-Diffraction Studies on the High-Temperature Oxidation of Iron-Aluminum Alloys. Tadayuki Nakayama. J. Japan Inst. Metals (Nippon Kinzoku Gakkai-Si), **23,** No. 1, 1-10 (1959). (In English.)

Electron-transmission diffraction studies of the high-temperature oxidation of iron-aluminum alloys containing Al 8.86, 19.3, and 28.6 wt.—% revealed that the oxidation-resistant surface-film contained varying quantities and compositions of α—Fe_20_3, γ—Al_20_3 and α—Al_2O_3 according to the aluminum content and the heat-treatment temperature. The excellent heat-resistant quality of aluminized steel is due to the formation of a γ—Al_20_3 film on the surface during the initial stage of oxidation below 1000 C. The heat-treated Fe-Al alloys containing aluminum 16.44, 19.30, 19.80 and 22.5% showed that the composition of the resistant surface-layer vary to a great extent according to the previous heat-treatment, the aluminum content, and the oxidation temperature.—MA. 19994

6.2.5, 6.3.10, 4.2.5, 3.6.2, 2.3.2

Stainless Steel and Related Alloys Versus Marine Environments J. C. Rowlands. Corrosion Technology, **6,** No. 12, 359-363 (1959) Dec.

Presents results of corrosion tests carried out in marine environments on ferritic and austenitic) and nickel and high nickel alloy (65 nickel-15 chromium-20 iron, 80 nickel-15 chromium-5 iron, 70 nickel-29 copper-1 iron). Tests included full-immersion and half-tide immersion in sea water and aerial exposure. Galvanic and crevice corrosion tests were also run under full immersion conditions. Stainless steels and high-nickel alloys were found to have good intrinsic corrosion resistance except in crevices. Austenitic stainless steels, especially 18/8 molybdenum, were least susceptible to crevice corrosion, but no alloy was found to be immune. It is emphasized that extreme care be taken to protect stainless steels from crevice corrosion in marine environments. Comments are made on progress in corrosion testing with particular reference to accelerated crevice corrosion test. Tables, 7 references.—INCO. 19991

6.3 Non-Ferrous Metals and Alloys—Heavy

6.3.5, 4.7

Compatibility of Niobium with Bismuth. G. W. Parry and L. W. Graham. Bull. Inst. Metals, **4,** No. 16, 125-126 1958).

A letter. A series of simple tests is described in which the solubility of niobium in bismuth at 600 C and its compatibility at 1000 C were determined. With niobium (iron 0.01-0.02, carbon 0.01, tantalum 0.1, oxygen 0.01%, traces of chromium and nickel) and bismuth (99.95%) and bismuth-magnesium (2000 ppm) it was found that although niobium did not suffer from intergranular attack under nominally static short-term tests in bismuth at 600 C, it had significant solubility. It is concluded that niobium is not suitable for containing liquid bismuth even under oxygen-free conditions above 600 C.—MA. 19371

6.3.19, 5.9.4, 5.8.3

Polarization Studies on Chromate-Treated Zinc. K. S. Rajagopalan and K. Balakrishnan. Current Sci. (India), **27,** No. 10, 387-388 (1958).

Mechanism of the inhibition by chromate treatment of the corrosion of zinc was examined. Polarization curves were obtained for untreated and chromate treated zinc in 0.001% sodium chloride

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solution, and for untreated zinc in a solution of 0.001% sodium chloride plus 500 ppm sodium chromate. Potentials were measured by the Poggendorf method, at c.d. from 0 to 250 μ amp/in.^{2}. It is concluded that inhibition results from the leaching into the corrosion medium of Cr^{6+} ions from the film formed on chromizing (a 0.00002-in. thick layer of hydrated $Cr_2O_3 \cdot CrO_3$). Both Cr^{6+} in solution and the Cr^{3+} ions remaining on the metal surface reduce corrosion.—MA. 18935

6.3.5, 3.7.2, 3.8.4

Development of Oxidation-Resistant Niobium Alloys. Harry P. Kling. Electrochem. Soc. Symposium on Technology of Niobium 1958, **1958,** 87-91. Available from John Wiley & Sons, Inc., New York.

Mechanism of the oxidation process is discussed and means of improving resistance are considered. Of the niobium alloys, those containing titanium, vanadium and molybdenum are most resistant to oxidation at 1000 C.—MA. 18740

6.3.15, 3.6.6

Chemical and Galvanic Corrosion Properties of Titanium Alloys. Charles B. Kenahan and David Schlain. Bureau of Mines, Report of Investigations No. 5423, March, 1958, 31 pp.

Chemical and galvanic corrosion properties of commercially pure titanium, a titanium-manganese alloy, a titanium-aluminum-tin alloy, 2 titanium-copper alloys and 2 titanium-aluminum alloys were studied. Tests were conducted in synthetic ocean water, tapwater, 1% sodium hydroxide, 5% ferric chloride, 5% ammonium sulfate, various concentrations of sulfuric and hydrochloric acids and 1% oxalic acids. All tests were carried out at 35 or 60 C, with a flow of air or helium. Dissimilar metals involved in galvanic-couples experiments were aluminum, magnesium and stainless steel. Electrode-potential measurements on single (uncoupled) specimens were also measured. Commercially pure titanium and the titanium-base alloys were generally quite similar in their resistance to corrosion, whether alone or in contact with a dissimilar metal. Contact with the titanium metals caused aluminum and magnesium to corrode by galvanic action. Coupling with stainless steel in sulfuric acid solutions decreased the normal corrosion rate of the titanium metals. Contact with aluminum in air-aerated oxalic acid and 0.1N sulfuric acid solutions resulted in cathodic corrosion of titanium and the titanium-base alloys; there were a few exceptions. The Cu alloys showed better corrosion characteristics than Ti in certain H_2SO_4 solutions but were more susceptible to chemical attack in some HCl solutions. 23 references. (auth)—NSA. 18739

6.3.14, 3.8.4, 3.5.9

Oxidation of Tin. Charles Luner. Trans. Am. Inst. Mining & Met. Engrs., Metallurgical Soc., **218,** 572-573 (1960) June.

Kinetics study of the oxidation of 99.78% pure tin foil in pure oxygen. Rate curves and logarithmic rate law plots are given for reaction in the temperature range 168-211.5 C at 8 mm. Hg pressure. 8 references.—RML 20032

6.3.20, 3.5.4, 8.4.5

A Study of the Effects of Fission Fragment Recoils on the Oxidation of Zirconium (thesis submitted to the University of Tennessee). William C. Yee. Oak Ridge National Laboratory. U. S. Atomic Energy Commission Pubn., ORNL-2742, May 13, 1960, 103 pp. Available from Office of Technical Services, U. S. Department of Commerce, Washington 25, D. C.

Effects of fission fragment recoils on the corrosion of zirconium in an oxygen atmosphere at 250 C were studied. Each of the experiments was conducted in an autoclave unit which contained four zirconium specimens and a source of fission fragments as required. Four runs—three of them with a source—were conducted in the ORNL Graphite Reactor at a maximum thermal neutron flux of 8.5 x 10^{11} neutrons/cm^2/second. The experiment was designed to provide for oxidation of specimens under conditions such as reactor radiations including fission fragment recoils, reactor radiation in the absence of fission fragment recoils, and no radiation. All other variables were maintained as constant as possible. The test specimens were maintained at 250 C while the control specimens operated at 265 C. The results of the analyses made on the zirconium specimens indicated that fission fragment recoils did physically affect the oxide film. Observations may be interpreted as the fission fragment recoils affecting the oxide film such that the oxide underwent a phase transformation and/or the oxide developed a fine particle size with random orientation. The limited data indicated that there was a greater weight gain in the zirconium specimens oxidized in-pile than those oxidized out-of-pile.—NSA. 20170

6.3.15, 3.4.8, 3.2.2, 1.2.2

Titanium as Material for Chemical Equipment. (In German.) H. van Kann and A. Pollanz. Werkstoffe u. Korrision, **11,** No. 8, 465-473 (1960) Aug.

Because of its favorable properties, titanium has very quickly passed through its first development stages and has meanwhile reached considerable production figures. Though the specific gravity of titanium is little more than half that of steel, its strength corresponds to that of high-grade steel. While these properties are of particular interest to aircraft production, the high corrosion resistance of titanium has led to a steadily increasing use of this metal as a material for chemical equipment. In this sphere, titanium is primarily used in cases where the corrosion problems encountered cannot be solved, or cannot be satisfactorily solved, by conventional materials. In many cases, the media concerned are those where the corrosion is caused by chlorine ions. This may cause the well-known phenomenon of pitting corrosion even with, e. g. stainless steel, while titanium can generally be regarded as absolutely corrosion-proof even in such media.

A number of illustrations of titanium components, some of them on a large scale, show the scope of applications for which this new metal is already being used and processed. Since the best processing conditions for titanium have now been worked out and the necessary machinery etc. is now available, the manufacture of titanium components and titanium linings is generally no longer very difficult. Special experience and care is required for the welding of titanium parts in order to prevent oxygen and nitrogen from reaching the melt and thus causing embrittlement. A number of hints of a structural nature that must be taken into account in using titanium should be of value to the designer.

Finally, it is shown on the strength of a number of examples that, in spite of its higher price compared with conventional materials, titanium often permits the most economic solution of a problem, all the more so as the prices of titanium have already shown a considerable decrease as the demand increased. 20169

6.3.19, 8.6.2

Zinc Corrosion Caused by Modern Detergents. (In German.) H. Stüpel and F. Koch. Werkstoffe u. Korroson, **11,** No. 1, 33-39 (1960) January.

An investigation has been carried out on the influence of soap and synthetic detergents on the corrosion of zinc, with special regard to the hardening constituent of the water. A description is given of a laboratory testing method which permits the determination of the corrosive effect of numerous detergent components and built-up detergents. Various substances are tested for their inhibiting effect in a standard syndet in hard and soft water, and the influence of repeated effects of fresh liquids on zinc is evaluated and discussed. With detergents of normal composition, polymer phosphates show the greatest intensity of corrosion which is merely (and clearly) exceeded by the effect of the combination of these phosphates with bleaching agents (sodium perborate). A working theory for the action of such combined corrosion is discussed. 19519

6.3.15, 6.3.20, 4.6.2

Hot Water Corrosion Resistance of the Hf-Ti Alloys. M. D. Carver and E. T. Hayes. Bureau of Mines. U. S. Atomic Energy Commission Pubn., USBM-U-60, November 10, 1955 (Changed from Official Use Only August 28, 1958), 10 pp. Available from Office of Technical Services, Washington, D. C.

Purpose of the investigation was to determine the corrosion resistance, relative to that of Zircaloy-2 and relative to that of others in the series, of each alloy of the Hf-Ti series comprising, in 10% steps, those in the range 10 to 90% Ti. Each alloy was made in the form of a 100 gram button which was rolled to sheet and then annealed at 900 C for 48 hr. and cut into corrosion specimens which were tested both in water at 680 F and in steam at 750 F, both tests being conducted according to standard practice established by the Zirconium Corrosion Committee for testing alloys of zirconium. The results indicated that relative to Zircaloy-2 the alloys of the Hf-Ti series of 10 to 30% titanium are not corrosion resistant in hot water or steam. They indicated also that for time periods up to 140 days in water and to 60 days in steam, the alloys of 40 to 50% titanium are generally comparable in corrosion resistance to Zircaloy-2 while those of 60 to 90% Ti are, in this respect, much superior. Over the entire range, corrosion resistance apparently improves with increasing titanium content although the amount of the improvement, for a given increase in Ti, becomes less at higher concentrations of Ti. (auth)—NSA. 19721

6.3.19, 4.3.2

Investigation of Zinc Corrosion in a Sulfuric Acid Electrolyte in the Presence of Impurities. (In Russian.) A. V. Pomosov, E. E. Krymakova and A. I. Levin. J. Applied Chem. (Zhur. Priklad. Khim.), **31,** No. 5, 734-742 (1958) May. 19396

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Topic of the Month

Experience With Blast-Furnace Slag As an Aggregate in Reinforced Concrete*

By C. P. LARRABEE(1) and S. K. COBURN(2)

Abstract

The role of blast-furnace slag as an aggregate in reinforced concrete is considered both in the light of present-day experience and in past erroneous reports. It was found that such slag actually is substantially inert insofar as any deleterious action toward steel is concerned. Several prestressed-concrete structures containing slag aggregate, in use for more than three years, showed every evidence of giving a potentially long and trouble-free service life.

4.2.3, 6.6.5

Introduction

BLAST-FURNACE slag is the nonmetallic product produced simultaneously with pig iron by the reduction of iron ore. It results from the continuous charging of limestone or dolomite, or a mixture of the two, accompanied by successive loads of coke and iron ore or iron-bearing agglomerate into a furnace, where calcination to the basic oxides of lime and magnesia is accomplished. These basic oxides react with the oxides of silicon and aluminum (present in the gangue and the coke) to form complex silicates and aluminosilicates. The resulting molten product floating on top of the molten iron is withdrawn from the furnace, allowed to cool, and processed for use in the construction industry. The chemical composition of slag falls within the following limits:

Lime (CaO)	36 to 45%
Silica (SiO_2)	33 to 42%
Alumina (Al_2O_3)	10 to 16%
Magnesia (MgO)	3 to 12%
Sulfur (S)	1 to 3%
Iron, manganese, boron, etc.	Traces

Because the metallurgist applies the term "acidic" to the oxides of aluminum and silicon in high-temperature silicate chemistry, considerable misunderstanding and misinformation has developed around the use of blast-furnace slag as a "safe" aggregate for contact with steel reinforcing bars in concrete structures. At ambient temperatures, however, the action of slag is alkaline, not acidic. An aqueous slurry of slag, for example, will develop a pH in the alkaline range. In fact, in extensive studies now being conducted at state universities and agricultural experiment stations, it has been demonstrated that slag is superior to limestone and dolomite as a neutralizing agent for acid soils. Furthermore, slag is used without an alkaline-enveloping medium (such as cement) in sewage-disposal beds, one of the major uses for slag. Here, steel riser tubes and feeder pipes are surrounded to a depth of 5 to 8 feet with crushed slag and exposed to a continuous supply of liquid sewage. There have been no reports of service failures after several decades of trouble-free performance in the many communities that operate such disposal systems.

Another basis for the erroneous contention that slag aggregate could ultimately be responsible for the corrosion of steel reinforcing bars is the presence of sulfur. At one time, it was believed that sulfide sulfur could be oxidized to sulfate with a resultant expansion in volume that would crack the concrete and expose the reinforcing bars to the entry of water. It was also believed that if the sulfur were converted to sulfuric acid, it would attack the steel. If these theories had been correct, then during the past 65 years numerous evidences of failure would have been observed and reported. A careful search of the literature failed to reveal any descriptions of concrete structures that deteriorated significantly as a result of having utilized blast-furnace slag as the aggregate. (It is well known that conspicuous or repeated structural failures of engineering equipment and structures are widely publicized, whereas consistently good service performance usually is recognized only at long and infrequent intervals. The latter remark is true of slag.)

Considerable evidence has been accumulated however, from a large variety of service applications demonstrating that blast-furnace slag in concrete is substantially inert insofar as any deleterious action toward steel is concerned. A cast-in-place concrete sewer of 4700 feet in MacDonald, Ohio, and an 8400-foot

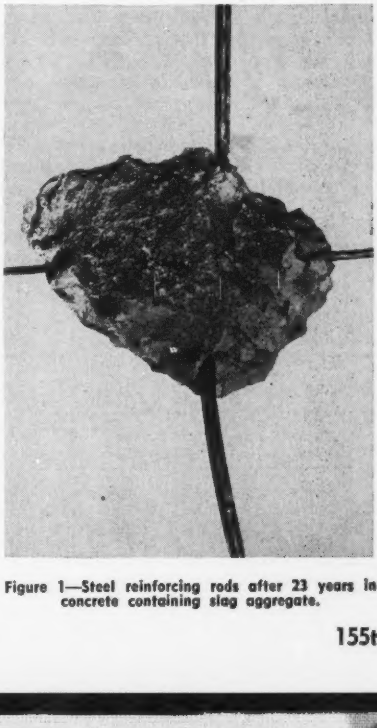

Figure 1—Steel reinforcing rods after 23 years in concrete containing slag aggregate.

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sewer in Atlanta, Georgia, are examples of structures utilizing slag aggregate in the concrete in an environment that could lead to eventual trouble if any component of the concrete structure failed. In 1937, tall, sloping revetments of slag concrete were built in Johnstown, Pennsylvania, to contain the periodic flood waters of the Conemaugh River. Other flood-control projects sponsored by the Corps of Engineers have specified slag as the aggregate. Numerous large buildings, bridges, and storage silos have been constructed with slag aggregate. Figure 1 shows an example of steel reinforcing rods embedded in a concrete pavement in 1935, removed in 1958, and found to be in excellent condition.

In every authenticated report describing service performance of slag-containing concrete, the authors commented on the need for a properly selected cement type as well as a correctly formulated concrete that should be added in sufficient quantity to provide a minimum cover of 2 inches over the reinforcing bars. Among the most important factors to consider in the formulation of sound concrete are the cement-water ratio, the use of appropriate additives, and air entrainment. The type of aggregate used normally plays an insignificant role up to this point.

Durability and Bond Strength

Service performance of concrete, however, is related to two other important factors: durability and bond strength. The long and exclusive use of slag in sewage trickling filter beds, where it is in contact with both acid and alkaline decomposition products and is subjected to frequent cycles of wetting and drying, and freezing and thawing, demonstrates its durability. Furthermore, slag has passed the sodium sulfate immersion test for determining the soundness of aggregate and the cyclic freezing and thawing tests for determining variations in properties of concrete as a result of variations in aggregate. These tests are described in the Book of Standards of the American Society for Testing Materials under designations C 88-56T and C 290-57T. As a result of these tests, blast-furnace slag compares favorably with natural limestone, sandstone, traprock, and gravel as an aggregate. It possesses an angular, pitted surface suggesting a porous structure that is noncommunicating. The sharp points and deep pits provide an increased surface area and numerous points for anchorage of the cement so that a bond of considerable strength results.

More recently, the idea of prestressed-concrete construction has been the object of many engineering investigations. In this application, the steel must possess great strength and any corrosion that might occur could be more damaging to the structure than in conventional reinforced-concrete structures. Several prestressed-concrete structures containing slag aggregate have been in use now for three years and exhibit every evidence of giving a potentially long and trouble-free service life.

In view of the above-mentioned favorable experience with blast-furnace slag, all the large specification-writing agencies have acknowledged the acceptability of slag as an aggregate material.

Any discussion of this article not published above will appear in June, 1961 issue.

Determining the Effect of Formulation On Physical Properties of Asphalt Mastic Coatings*

By *LYLE R. SHEPPARD*

Abstract

Field and laboratory tests were made to determine the characteristics of asphalt mastic coatings which may affect their performance on pipe lines as a corrosion protection device and as a weighting agent. A Blunt Needle Penetration Test was developed to aid in control of the quality of finished mastic coatings. This test proved quite sensitive to both binder quality and the ratio of binder to aggregate. Laboratory measurements were made of such physical properties as binder softening point and penetration, bulk density, adhesion, cohesion, apparent voids, blunt needle penetration, and temperature characteristics. Purpose of tests was to determine the mastic quality and the influence of various mixtures on physical properties of the mastic. A number of extensive outside discussions are included together with the author's replies.

5.4.5, 8.9.3, 2.3.7

Introduction

AN ASPHALT mastic is an external pipe line coating composed of aggregate (usually sand, crushed limestone and fibers), bound together with a blown asphalt. Often heavy minerals such as barite or iron ore are incorporated in the aggregate to give the coating greater bulk density. The mixture of materials is heated until the blend is fluid enough to be extruded onto the pipe.

Asphalt mastic coating is designed both for corrosion protection and for weighting of under-water pipe lines. It serves the same purpose as a coating system of hot enamel covered with concrete. However, mastic has advantages over the concrete system, of application, handling, and additional coating thickness for corrosion protection. The common disadvantages of both systems are yard or rail-head application and field coating of joints after welding.

Probably the best tests ever conducted on pipe line coatings were those made from 1930 to 1940 by the Bureau of Standards and the American Petroleum Institute. An asphalt mastic was one of the better coatings surviving these tests. Table 1 shows the comparative results of these simultaneous tests of mastic, asphalt enamel, and coal tar enamel. It will be noted that the average of maximum pit depths under the mastic coating was extremely shallow (0.12 mils) as compared to the average of maximum pit depths under the enamel coatings (21.77 mils and 12.10 mils). The average of maximum pit depths on uncoated pipe at all the test sites for mastic and enamels was approximately the same (63.5 mils to 69.0 mils). The ratio of the pit depths on coated to uncoated pipe was in favor of mastic (0.17) over enamels (9.42 and 12.89), even when differences in coating thicknesses were taken into consideration. Higher percentages of unaffected or unetched pipe under the mastic indicated that it protected the pipe from corrosion better than the enamels did.

Actual service experience with asphalt mastic has justified the good rating obtained in the above tests. A 65 mile section of the lower Mississippi River pipe line, mentioned in this paper, has an average current density requirement for cathodic protection of 0.0014 milliamperes per square foot after six months under-water burial. Another mastic coated line in central Louisiana, laid in 1942, has an average current density requirement of 0.011 milliamperes per square foot at the present time. Other lines, laid at approximately the same time with conventional enamel coatings, had to be reconditioned and recoated after fifteen years of service. From study of these tests and experiences there can be no doubt of the economic effectiveness of asphalt mastic pipe line coatings.

About the Author

LYLE R. SHEPPARD at the time of his death in December of 1960, was senior engineer in corrosion research with Shell Pipe Line Corp., Houston, Texas. He held BS and MS degrees from the University of Houston and was a charter member of NACE. Mr. Sheppard had been engaged in pipe line corrosion control work since 1932.

Test Programs

During the last half of 1958 a field and laboratory test program was carried out on an asphalt mastic coating applied to a 20-inch OD oil pipe line from the mouth of the Mississippi River to a refinery just north of New Orleans, Louisiana. In addition, a number of heavy aggregate asphalt mastics were laboratory tested over a period of two years ending early in 1959. The purposes of these test programs were to determine the mastic quality and the influence of various mixtures on physical properties of the mastic.

The first test program was made on asphalt mastic designed to meet the Asphalt Institute Grade II Specifications. It consisted of tests conducted at the application yard and in the laboratory.

A modified penetration test was developed in the laboratory for use at the application yard on mastic coated pipe. This test was called the "Blunt Needle Penetration" test because of the shortened and blunted needle used in the penetrometer. The value of the penetration used is the average of eight individual penetration tests on the mastic. Such an average is reproducible within ASTM tolerances (± 2 dmm + 2 percent of reading). The blunt needle penetration is mostly influenced by differences in binder softening points or penetrations, and difference in proportioning of the binder and aggregate. The blunt needle penetration test, consequently, was used for quality control at the application yard. Every fiftieth joint of coated pipe was tested.

Equipment was also available at the application yard to make tests of asphalt penetration and aggregate specific gravity. These tests were not normally run unless the blunt needle penetration tests showed the process to be out of control.

Daily samples of the binder and of the finished mastic from the yard-tested joints were sent to the laboratory for

TABLE 1—Bureau of Standards—API Coating Tests (1930-1940)*

	Test Symbol and Coating Type		
	O, Asphalt Mastic	R, Asphalt Enamel	T, Coal Tar Enamel
Coating Thickness—mils	519	143	351
Average-Maximum pit depth—(coated)—mils	0.12	21.77	12.10
Average-Maximum pit depth—(uncoated)—mils	69.0	63.7	63.5
Number of test sites	10	10	8
Ratio pit depth-coated to uncoated-percentage	0.17	34.18	19.06
Ratio pit depths-Adjusted to 519 mils-percentage	0.17	9.42	12.89
Total feet inspected—100 percent	213	208	175
Unaffected—percentage	72.3	20.2	14.9
Rusted—percentage	18.3	9.1	14.3
Metal Attack—percentage	6.6	20.2	33.1
Pitted—percentage	2.8	50.5	37.7

* These data taken from **"Underground Corrosion"**, Tables 74 and 75, pp. 152-158, 1945, Bureau of Standards Circular C450.

★Submitted for publication August 24, 1959. A paper presented at a meeting of the South Central Region, National Association of Corrosion Engineers, Denver, Colorado, Oct. 12-15, 1959.

further evaluation. The laboratory evaluation included tests on the mastic components before combination, the components separated from the finished mastic, and the finished mastic. Binder softening point, penetration, mastic proportioning, bulk density, adhesion, cohesion, apparent voids, blunt needle penetration, and temperature characteristics were determined in the laboratory on the daily composite samples taken throughout the period of application. No exposure tests were made. Tests at low temperature were not made because low temperature operation was not considered to be a problem in the lower Mississippi River Valley. Tests used are described in the Appendix.

The second series of tests were made on heavy aggregate asphalt mastics. Laboratory tests only were made on these materials. In addition to the tests described, the binder was extracted from the finished mastic and tested for comparison with the binder before being incorporated into the mastic. Extraction was made by solvent at low temperatures, never greater than 265 F, and under vacuum.

Test Results

These two test programs must be considered individually, though several common conclusions can be drawn from both. The test program on the Grade II mastic is believed to be the first attempt at quality control on this type of coating by a purchaser. Several times during application, physical tests detected a decline in mastic quality which was always corrected before off-specification conditions were reached. In general, the quality of the product was very uniform and no difficulty was encountered in meeting Asphalt Institute specifications.

The ranges of the test results on the Grade II mastic were so narrow that little information concerning the effects of various formulations on the mastic properties could be determined. However, the few test extremes were studied and some general conclusions were determined from them.

Program 1

In the first test program, the aggregate was not sampled nor tested daily, since sand from one source was the main ingredient. Daily tests of bulk density and size gradation of the production aggregate mixture should be made when heavy aggregate is used with the sand.

Table 2 gives a summary of the test results on 70 daily composite samples of Asphalt Institute Grade II mastic as produced during regular application. The maximum and minimum numerical test values do not necessarily represent the same sample. The sample with the maximum softening point temperature would most likely be the same as that with the minimum penetration at 77 F.

Asphalt binder softening point and penetration were quite uniform at approximately the mean specifications for Grade II mastic (softening point 187.5 F, penetration 16.0 dmm). Softening point range was only 15.5 F compared to 25 F allowable. Penetration range was 3.3 dmm compared to 2.0 dmm allowable. One batch having 13.7 dmm penetration caused this wide range. This batch was not rejected because the mastic made with it had greater adhesion and cohesion, and lower temperature susceptibility than that required in Grade II specifications. The range of penetration values permitted in the specification for Grade II mastic is narrower than for any other grade and is unrealistic in terms of the variability allowed in measuring penetration. It seems likely that the specification should be widened to allow 14 to 18 dmm penetration, which would make for greater ease in manufacture with no real sacrifice in properties of the mastic.

All batches of binder had an average Penetration Index of 2.45 with a narrow range of 0.75. Average Penetration Index of Grade II is 2.60 with a range of 1.90. A Penetration Index of 2.60 indicates that the asphalt has definite elastic properties.[1] This means that it has good resistance to "cold flow" and to deformation by continued pressure. Naturally, the addition of aggregate increases this resistance.

Percentage of binder by weight in the mastic averaged 13.14 percent and had a range of 11.22 percent. While there is no Asphalt Institute Specification for percentage of binder, the mastic manufacturers normally try to keep the values between 12 percent and 14 percent for Grade II mastic. No reason for the observed wide range was determined. It certainly was not caused by non-uniformity of binder. The wide ranges in some of the mastic properties were due to the lack of close control over the percentage of binder.

Bulk density and apparent voids of the mastic as received varied inversely with the percentage of binder. Average values for these two properties were 131.4 lbs. per cubic foot and 3.5 percent volume, respectively, with corresponding ranges of 5.7 lbs. per cubic foot and 4.3 percent volume. The variations in these properties could be traced to variations in volume percentage of binder in every case. Within the narrow ranges tested, binder softening point and penetration had little effect on these two related mastic physical properties. It is possible that mixing time and extrusion speed and temperature also affect these properties, though these effects were not tested.

Laboratory tests on the 70 batches summarized in Table 2 showed that mastic adhesion to steel and cohesion varied directly with binder softening point and inversely with binder penetration and percentage. Of these, binder percentage had the greatest effect. It was difficult to evaluate the effect of each because both softening points and percentage of binder varied simultaneously. However, the following three batches illustrate the effect of different softening points on adhesion:

Softening Point, Degrees F	Percent w Binder	Adhesion, psi
185	11.4	231
187	11.4	245
190	11.4	268

The effect of percentage of binder on adhesion is illustrated by the following three batches:

Softening Point, Degrees F	Percent w Binder	Adhesion, psi
185	8.3	275
185	15.3	236
185	19.6	195

Cohesion of these samples was within the same values and ranges as adhesion.

Even the lowest values of adhesion and cohesion were well above the minimum Grade II specifications of 75 psi for ad-

TABLE 2—Summary of Laboratory Tests on Seventy Batches

(Asphalt Institute Grade II Mastic)

Property:	Specification	Minimum	Average	Maximum	Range
Asphalt Binder:					
Softening Point—°F	175—200	180.0	186.0	195.5	15.5
Penetration at 77 °F, 100 gm-5 sec, dmm	15— 17	13.7*	15.8	17.0	3.3
Penetration Index (Calculated)		2.14	2.45	2.89	0.75
Mastic as Received:					
Bulk Density—lbs/cu ft	....	128	131	134	5.7
Ash—Percent w	....	71	76	81	9.9
Binder—Percent w	....	8.3	13.1	19.6	11.2
Binder—Percent v	....	17.2	26.5	39.7	22.5
Mastic Pressured to 1500 psi:					
Bulk Density—lbs/cu ft	....	132	136	139	6.9
Adhesion—Primed—psi	75	193	232	279	86
Cohesion—psi	150	179	257	300	121
Apparent voids—Percent v	....	1.1	3.5	5.4	4.3
Pen.—1300 gm-30 sec—77 °F, dmm	....	4.5	8.5	15.5	11.0
Pen.—1300 gm-30 sec—115 °F, dmm	....	15.5	59.0	149.0	133.0
Temperature Coefficient	....	0.014	0.022	0.026	0.011

* This was one composite sample of 70 tested. It was not rejected because the mastic made from it had superior properties in the other tests, and it actually almost met specifications for Grade III.

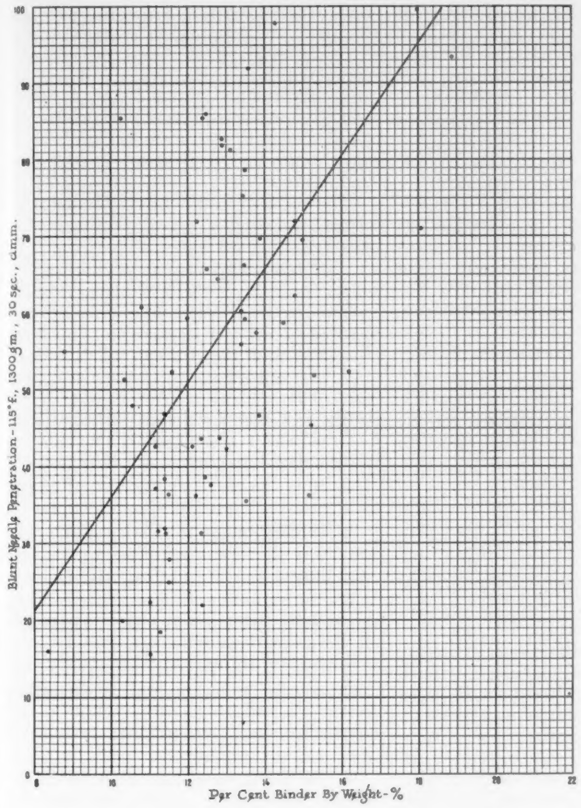

Figure 1—Penetration versus percent binder for Grade II mastic.

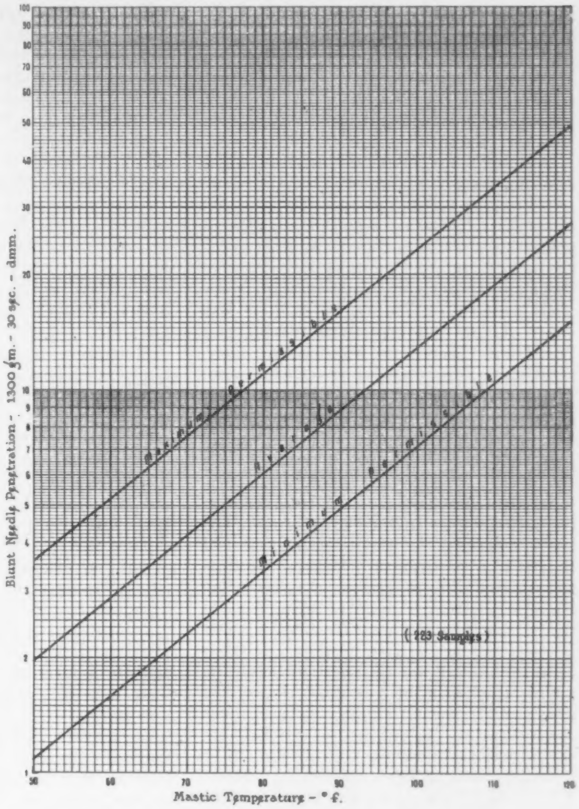

Figure 2—Penetration versus temperature for Grade II mastic.

hesion and 150 psi for cohesion. The wide ranges of 86 psi and 121 psi could be made narrower by closer control of binder percentage. Cohesion was greater than adhesion with four exceptions in 70 samples. Each of these four exceptions had an abnormally high percentage of binder.

Temperature susceptibility of mastic varied directly with binder percentage, and inversely with binder softening point. This is consistent with the behavior of filled bitumen enamels. Temperature susceptibility coefficient was calculated as follows:

$$\text{coefficient} = \frac{\log P_{115} - \log P_{77}}{115 - 77}$$

where P_{115} = blunt need penetration at 115 F.
P_{77} = blunt need penetration at 77 F.

It averaged 0.0222 with a range of 0.0114. Temperature susceptibility was the mastic property most sensitive to changes in quality of materials and their proportioning.

Figure 1 illustrates the effect of proportioning on mastic penetration at 115 F. The straight line was fitted to the data by method of least squares. Unfortunately, the data shown refer to binder mixtures in which the softening point varied as well as binder percentage, thus giving widely scattered penetration values.

TABLE 3—Laboratory Test Results on Heavy Mastic Samples

Sample No. Date	None 3/57	436–57 8/57	456–57 1/58	30–59 2/59
Binder as Received (Without rubber)				
Softening Point, °F	......	154.5	......	165.5
Pen. @ 77 °F—100 gm-5 sec, dmm	......	26.5	......	21.5
Pen. Index (calculated)	......	+1.00	......	+1.53
Extracted from Finished Mastic (including rubber)				
Softening Point, °F	134	159	139	170
Pen. @ 77 °F—100 gm-5 sec, dmm	48.0	28.5	39.0	29.0
Pen. Index (calculated)	+0.24	+1.53	+0.33	+2.55
Mastic From Pipe as Received				
Bulk Density—lbs/cu ft	157	135	167	190
Percentage Ash, Percent	86	81	85	90
Percentage Binder (weight), percent	11.0	10.4	10.1	6.7
Percentage Binder (volume), percent	24.6	21.6	25.3	19.4
Mastic Pressured to (1500 psi)				
Bulk Density—lbs/cu ft	170	137	166	201
*Adhesion—Primed—psi	29	29	107	164
Cohesion—psi	189	218	186	286
Pen. @ 77 °F—15 sec-1300 gm, dmm	13.5	11.5	11.5	5
Pen. @ 77 °F—30 sec-1300 gm, dmm	18	14	20	6
Pen. @ 77 °F—60 sec-1300 gm, dmm	24	16	30	7
Pen. @ 115 °F—30 sec-1300 gm, dmm	530	400	760	18
Temperature Coefficient	0.038	0.039	0.042	0.013
Apparent Voids Percent v	7.8	1.4	+0.6	5.3

* Oil Primed—This practice has been discontinued because of difficulty of control.

Program 2

The mastic tested in the second program differed in formulation from that in the first program. Test results are shown in Table 3. The asphalt binder was softer (Penetration 28.3 to 48.0 dmm) and had lower softening points (134.0 to 170.3 F). Also, a small amount of rubber emulsion (between 1½ and 3 percent of total binder) was added to the binder. Heavier aggregate was used in three of the four samples tested. Percentage of binder did not cover as great a range (6.65 to 10.96 percent weight) as those in the first series of tests (8.34 to 19.56 percent weight).

The effect of the rubber addition to the binder was to widen the temperature range; that is, to increase both the penetration (by 2.0 and 7.7 dmm) and softening point (by 4.2 and 4.8 F) of the asphalt binders. This means that the mastic made from the rubberized binder will operate successfully at both lower and higher temperatures than that made from the original binder. These two effects cannot be obtained simultaneously by asphalt processing or addition of plasticizing oils.

Two of the binders were close to the specifications for Grade I mastic (softening point 150-175 F, penetration 21-25 dmm) and two were softer, having lower softening points than those for any grade of mastic as published by the Asphalt Institute. Three of the binders, those with penetration indices of 2.00 or less, were of the type which normally give "cold flow" and poor temperature characteristics. Blunt needle penetration tests of the produced mastics made from these three binders showed exceptionally high penetration values at 115 F, which is consistent with the low penetration indices.

Barite was used as the additional weighting agent in the three heavy mastics. According to the producer of this mastic it is possible to attain a bulk density of 220 lbs. per cubic foot by using barite. If either ilmenite (Sp. Gr. = 4.5 — 4.9) or magnetite (Sp. Gr. = 4.9 — 5.2) were used instead of barite (Sp. Gr. = 4.3 — 4.6) as the weighting agent, it should be possible to produce a mastic having a slightly greater bulk density. It also seems likely that because of the greater hardness of ilmenite (H = 5.0 — 6.0) and magnetite (H = 5.5 — 6.5) as compared to barite (H = 2.5 — 3.5), these aggregates would have sharper edges and thus increase the cohesive and compressive strength of the mastic, because of the increased internal resistance to flow under stressing. The producer has presented one possible processing disadvantage with the use of these two materials—excessive wear of the extrusion dies because of the greater hardness. One operating disadvantage has been presented—the mastic would have less electrical resistance because both of these iron ores have greater electrical conductance than barite. This should not be a problem at low voltage since each particle of aggregate would be completely covered with binder if the mastic were properly proportioned and mixed. Thus, the electrical conductance of the binder would be the controlling factor at low voltages.

There was considerable variation in the binder softening points and penetrations in this series of tests resulting in wide variations in mastic properties. There was only slight variation in percentage of binder.

Bulk density was purposely varied from 134.78 to 190.32 lbs. per cubic foot by the addition of various amounts of barite. There did not appear to be any great effect on other mastic properties as a result of increasing bulk density.

Apparent percentage of voids varied more than in the first series of tests. This probably was due to variations in extrusion conditions as well as in percentage of binder and binder softening point and penetration.

It is possible that the adhesion to pipe is greater than the laboratory test values (28.6 to 164.3 psi) indicate. This is at least partially due to the pipe's becoming slightly heated by passage through the heated extrusion chamber during application. Laboratory tests showed that heating the steel improved adhesion. Improper laboratory techniques with oil priming may have contributed to the lower laboratory adhesion test values.

Cohesion varied rather widely (185.7 to 285.7 psi). This was due to variations in both percentage and type of binders. Low binder softening point and high penetration were associated with low cohesive strength. An excess of binder also gave low cohesive strength.

Resistance to penetration at higher temperatures was low on three of the four samples tested. This was mainly due to the low binder softening points and high penetrations. Slightly greater than normal percentages of binder also increased mastic penetrations.

Certain conclusions were drawn from both test programs. One point realized during this test program was that when designing mastics of varying bulk densities the binder percentage should be based on volume rather than weight for optimum mastic properties. The percentage of binder by volume varies only slightly in making good mastic of various bulk densities, because mastic bulk density is increased by increasing aggregate bulk density, not amount. There is a tendency to have too much binder in heavier bulk density mastic. This results in low adhesion and cohesion, together with increased temperature susceptibility. The optimum mastic properties in both test programs appeared to be attained with 21.5 percent to 22.5 percent of binder by volume regardless of the weight of the aggregate. A mastic with lower percentage of voids can be made by using more than this amount of binder, but the temperature, loading, and cohesive properties will be adversely affected by such proportioning. If less than this amount of binder is used, "dry spots" or aggregate not covered with binder result, adversely affecting the above mastic properties.

Because blunt needle mastic penetration was quite sensitive to changes in quality and proportioning of materials, it is suggested as a quality control during application at the plant. In the 70 batches tested in the first program no changes occurred in the quality of the mastic without being reflected in the blunt needle penetration tests. Figure 2 is a control chart of penetration versus mastic temperature based on the yard test results of 223 joints tested during application. It was established after approximately fifty joints had been tested, and little change occurred afterward with more test data. The minimum and maximum values lines were set at one standard deviation from the average value line because all joints having values beyond them were found to have some less favorable physical properties. This chart was necessary because mastic temperatures on the pipe could not be controlled, only measured. It should be recognized that this chart applies only to Grade II mastic. Figure 1, from laboratory tests, appears inconsistent with Figure 2. This probably is due to differences in testing techniques, especially of temperature measurements. The yard applied mastic would have a surface which is cooler and harder than the material beneath the surface, resulting in apparently harder mastic at the same temperature.

The relationships between binder properties and mastic properties are summarized in Table 4.

Summary

The external pipe line coating tests conducted by the American Petroleum Institute and the Bureau of Standards and subsequent service experience indicate that asphalt mastic is one of the better coatings developed for pipe lines.

Conclusions drawn from the two test programs completed in 1958-59 were:

1. Blunt needle penetration of mastic is a practical test for quality control.
2. Proportioning of binder and aggregate was the operation least under control during mastic production.
3. Proportioning design should be done by volume rather than weight regardless of the bulk density of the mastic.
4. Binder softening point directly affects mastic adhesion and cohesion; inversely it affects mastic blunt needle penetration and temperature susceptibility.
5. Binder penetration directly affects mastic bunt needle penetration and temperature susceptibility; inversely it affects mastic adhesion and cohesion.
6. Percentage of binder directly affects mastic blunt needle penetration and temperature susceptibility; inversely it affects mastic bulk density, percentage of voids, adhesion, and cohesion.
7. Properly formulated and applied mastic has greater cohesion than adhesion.
8. Further work is necessary on the effects on mastic properties of extrusion speed, application temperature, and cooling after applications.

Acknowledgment

Cooperation of the H. C. Price Company and the Timcoat Corporation in making these tests is appreciated. The help and constructive criticisms of the Technical Development Laboratory staff, and the permission of the Management of the Shell Pipe Line Corporation for

TABLE 4—Relationship of Binder and Mastic Properties

	BINDER		
MASTIC	Softening Point	Pen. at 77 F	% By Wt.
Bulk Density (From Pipe)			Inverse
Adhesion to Steel........	Direct	Inverse	Inverse
Cohesion........	Direct	Inverse	Inverse
Percentage voids........			Inverse
Blunt Needle Pen. at 77 F	Inverse	Direct	Direct
Blunt Needle Pen. at 115 F.	Inverse	Direct	Direct
Temperature Susceptibility	Inverse	Direct	Direct

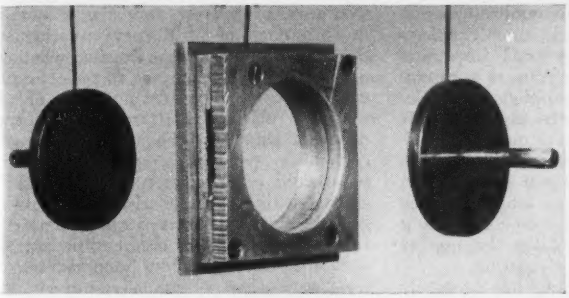

Figure 3—Adhesion plates and mold.

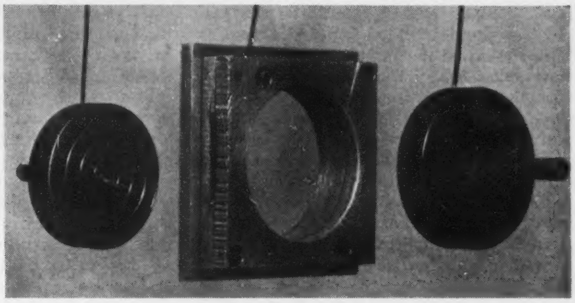

Figure 4—Cohesion plates and mold.

publication of this work are gratefully acknowledged.

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1. J. P. Pfeiffer. The Properties of Asphaltic Bitumen. Elsevier Publishing Co., Inc., New York, N.Y., 1950, p. 168.

APPENDIX 1—Test Methods

On Asphalt Binder

1. Softening Point—ASTM—D36-26
2. Penetration—ASTM—D5
3. Calculation of Penetration Index

$$\text{P. I.} = \frac{20\Delta T - 500\ (\Delta \log \text{Pen.})}{50\ (\Delta \log \text{Pen.}) + \Delta T}$$

where:

P. I. = Penetration Index
ΔT = Temperature differential, (Softening point temperature less 77 F), °C
Δ log Pen. = log Penetration Differential (log 800 less log of Pen. at 77 F), log dmm.

NOTE: The above equation is taken from J. Ph. Pfeiffer, *Properties of Asphaltic Bitumen*, pp. 166-167, 1950, Elsevier Publishing Co., Inc., New York. It assumes that the penetration for 5 sec. with 100 gm. loading at the softening point temperature is 800 dmm.

On Mastic As Received

1. Bulk Density—ASTM—D71-27
2. Percentage Ash—ASTM—D271
3. Percentage of binder (by weight)—This is calculated from ashing tests (ASTM—D271) of the finished mastic and the total aggregate, without binder.
4. Percentage of binder (by volume)—By calculation from percentage by weight.

On Pressured Mastic

Fifteen hundred pounds per square inch pressure was selected for reforming the mastic test specimen because that value was used in the Asphalt Institute Specifications. In preliminary testing it was found that pressures greater than this value did not increase bulk density nor reduce apparent voids. Consequently, the mastic physical properties for any specific mixture obtained at this pressure were considered to be the maximum obtainable.

1. Bulk Density—ASTM—D71-27

2. Adhesion—Test developed by Shell Pipe Line Corporation. Two steel plates, 3" dia. x 9/16" thick, each having a 3/8" x 2" rod attached to the center, are uniformly primed on the flat surfaces opposite the rods. Figure 3 shows the adhesion plates and mold. The primer is allowed to air dry until not sticky to touch or approximately two hours. One plate is placed in one end of a cylindrical mold. The mold surfaces are covered with a thin film of glycerine to prevent mastic adhesion to them. Plates, mold and mastic are then heated to approximately 325 F. (When rubber emulsion is used in the binder this temperature is reduced to 275 F so that the rubber will not be damaged by the heat.) Enough heated mastic to compress to 5/8" thick cylinder 3" in diameter is added. The second plate is pressed onto the hot mastic at 1500 psi or a total load of 10,600 lbs., and held in the mold for one minute. Pressure is released and the entire assembly is cooled immediately to approximately 100 F by water. The two plates with the mastic between them are then pressed from the mold.

After air cooling in a horizontal position for 12 hours at approximately 77 F, the two plates are pulled in a tensiometer at 0.5 inch per minute until failure. The test is made in duplicate.

3. Cohesion—Test developed by Shell Pipe Line Corporation. The cohesion test is similar to the adhesion test except that the specimen plates are designed so that the adhesion area is greater than the specimen cross-sectional area, thus assuring a fracture in the mastic rather than at the plate surface. Figure 4 shows the cohesion plates and mold.

The test specimens are prepared in the same manner as the adhesion test specimen. The test loading rate and temperature is the same as for the adhesion test. The test is made in duplicate.

The mastic adhesion and cohesion can be compared directly because the specimens cross-sectional areas are the same.

4. Percentage of Voids—Percentage of voids is calculated from the mastic bulk density as received and as repressured to 1500 psi. This assumes that the maximum bulk density for any specific mixture is obtainable at 1500 psi.

5. Blunt Needle Penetration—Test developed by Shell Pipe Line Corporation—This test is based on the statistical principle that enough individual tests, with wide variations, will average repeatedly within close tolerances. The test is a modification of ASTM—D5. A standard penetration needle is shortened to 15 mm, and ground to a 43 degree conical point. The test time is increased to 30 seconds, and the loading to 1300 gm. Figure 5 shows a portable model penetrometer modified to set on coated pipe, and with the additional loading weight and blunt needle in position.

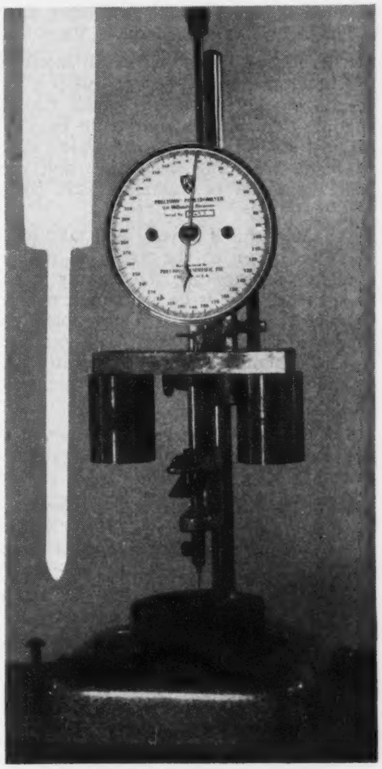

Figure 5—Portable model penetrometer modified to set on coated pipe. Silhouette cutout at left is a magnified view of point.

In the laboratory the pressurized mastic coupons prepared for the adhesion test are used for this test. They are brought to testing temperature and maintained there for two hours before testing. Test temperatures are 77 F and 115 F. Six individual tests are made and averaged at each temperature. Test reproduction averages (of six) are within ASTM tolerance (1 dmm + 0.01 of penetration).

When this test is used for quality control in the application yard, eight readings are averaged, and the temperature taken of the mastic on the pipe. Figure

2 is a control chart of penetration vs. mastic temperature for Grade II mastic. This chart can be made after fifty joints have been tested and should not change significantly with further tests. The limits were set at one standard deviation because all batches having unfavorable physical properties had penetrations outside those limits.

6. Temperature Susceptibility Coefficient*—The coefficient of temperature susceptibility is calculated from the blunt needle penetration tests as follows:

$$\text{Coefficient} = \frac{\text{log of Pen. @ 115 F} - \text{log of Pen. @ 77 F}}{115\text{-}77}$$

Comments by Raymond F. Hadley, Sun Pipe Line Co., Philadelphia, Pa.:

Mr. Sheppard has prepared an interesting and informative paper relating to certain characteristics of asphalt mastic coatings. There are, however, a number of suggestions or comments which may be made regarding this paper and the general subject of asphalt mastic coatings.

It is doubtful if anyone will take serious exception to the premise that a method for checking the quality of a product, whether it be an asphalt mastic or any other coating, is a desirable step in obtaining a more suitable product from the manufacturer or applicator.

There are, however, two questions to be asked:

1. Do existing data indicate a need for a more thorough check of product quality?

2. Is the present method of checking quality inadequate and if so, is the method proposed by Mr. Sheppard more suitable?

With reference to the first of these questions Mr. Sheppard in his paper mentions that the current density requirements for an asphalt mastic coated line laid in 1942 at present has an average current density requirement of 0.011 milliamperes/sq. ft. and that another line similarly coated has a current density requirement of 0.0014 milliamperes/sq. ft. after six months of underwater burial. Our experience with asphalt mastic coatings fully verifies the figures reported by Mr. Sheppard, for an asphalt mastic coated section of line applied in 1944 requires approximately 0.010 milliamperes/sq. ft. at the present time and our most recent application of asphalt mastic coated pipe installed in 1956 required but 0.0015 milliamperes/sq. ft. three years after installation.

Generally speaking, our experience indicates that, for all of our lines protected with asphalt mastic coatings, the maintained electrical resistance of this type of coating is so much greater than the electrical resistance of the other types of coatings we have used that they constitute a family of electrical resistance curves of an entirely different order of magnitude.

In the application of the coatings referred to above, at least on our system, and it is presumed the same applies to the earlier applications on Mr. Sheppard's system, no physical inspection other than visual and electrical were employed at the time of application to determine the quality of the asphalt mastic coating. Inasmuch as the current densities required to provide cathodic protection continue to remain insignificant, even after periods of as long as fifteen years, the question may be asked, is it really necessary to monitor the quality of this particular type of coating.

It seems to us, since we are primarily interested in a maintained high electrical resistance of the asphalt mastic coating for long periods of time, the most pertinent data relating to the situation have not been developed (namely, how does the resistance of the asphalt mastic coating change as a function of time in various environments with different percentages of asphalt content in the mastic).

Dr. J. F. Putnam,[1] in his report prepared for the 1937 corrosion conference at the National Bureau of Standards, which report incidentally is the most comprehensive relating to the asphalt mastic pipe coating which has come to our attention, shows that for asphalt mastic coating the maximum tensile strength of the mastic occurs at approximately 8.1 percent asphalt content (by weight) which corresponds very closely to the 8.7 percent asphalt required to just fill the mastic void volume. This is equivalent to a 20 percent void space in the mastic on a volume basis.

Nowhere has it been conclusively demonstrated that the maintained electrical resistance of the mastic coating suffers as a result of increasing the asphalt content of the coating from the strongest, most dense mastic (which is difficult to apply) to a less dense mastic containing as much as 14 percent asphalt, but which can readily be applied to a pipe. We believe that these data should be developed before it can really be said that there is a need for more stringent quality control.

In regard to the second question relating to the present method of checking the quality of the asphalt mastic coating, the Product Lines Department of the Sun Pipe Line Company have used only what could be termed a qualitative check of the coating rather than the more complete quantitative tests such as those described by Mr. Sheppard. Qualified personnel from our Corrosion Department, who have had considerable experience in the application of the asphalt mastic coatings believe it possible to adequately judge the quality of an asphalt mastic coating by visual inspection as the coating leaves the extrusion nozzle.

Although the writer believes that experience is an invaluable asset to the coating inspector, the development of suitable quantitative tests for the coating nevertheless could also be extremely helpful in insuring the application of a coating of optimum quality, provided such tests are proven reliable and can be made rapidly at little expense to the company.

In regard to the modified penetration test developed by Mr. Sheppard, it would be of interest to learn, following the approach of McBurney[2] relating to the indentation of asphalt tile, whether or not an effort was made to collect data which would enable development of an equation relating penetration to time for different loads. In the absence of such correlation perhaps a sphere, cylinder, paraboloid of revolution or some other geometrical shape could be advantageously used to measure penetration rather than by using the modified blunt needle described by Mr. Sheppard.

The extreme scatter of data shown in Mr. Sheppard's Figure 1 rather discourages the use of a blunt needle and suggests that reproducibility by this particular measurement technique leaves much to be desired despite the fact that Mr. Sheppard has very properly fitted a curve to the data by the method of least squares. Furthermore, the observation that one must obtain an average of six individual readings to obtain a reproducible average within ASTM tolerances further indicates the need for additional work in the development of a suitable penetrometer. It would seem to us, as a matter of fact, very unlikely that a blunt needle penetrometer developed for the measurement of highly viscous fluids or viscous fluids fortified with fillers, having particle sizes small compared to the point diameter of the penetrometer, would be suitable for determining penetration of a mastic which acts as a virtual solid due to its content of finite size cubical particles.

Another important point requiring consideration is the rate of loading applied to the penetrometer, for Putnam has shown that the strength of an asphalt mastic coating with different percentages of binder asphalt is very definitely a function of the loading rate.

It is also noted that the mastic coupons prepared for the adhesion tests were used in Mr. Sheppard's laboratory blunt needle penetration tests. It is the thought of the writer that, the use of pressurized coupons could reasonably be expected to provide more uniform data from the penetration tests. He feels, nevertheless, that by using such pressurized coupons another variable, which must also require evaluation, has been added to a situation already over-complicated, since our real objective is quality of mastic applied to line pipe.

The adhesion and cohesion tests reported by Mr. Sheppard are extremely interesting. Here again, though, the pressurizing technique may result in additional complication when interpreting the data in regard to how the same may influence a coating applied to line pipe.

In Mr. Sheppard's paper an effort also has been made to evaluate some of the characteristics of the more dense asphalt mastic coatings. In this connection the writer has only one comment to offer, namely, that to obtain relatively high negative buoyancies through the use of more dense mastic as required for some lines, problems conceivably could arise relative to maintenance of mastic adhesion and cohesion when the required mastic thicknesses were applied to line pipe.

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* Herbert Abraham. Asphalts and Allied Substances. D. Van Nostrand Company, Inc., New York, p. 1005, 1945.

There are certain advantages to be gained in the protection of the underlying asphalt mastic coating as a result of obtaining the required increased weights by the use of high density concrete coatings applied over the mastic coating. As a matter of fact, we have in certain instances utilized high density concrete coatings over asphalt mastic coatings for physical protection of the mastic coating under certain severe environmental conditions where negative buoyancy was not even required.

References

1. Joseph F. Putnam. Asphalt Mastic Pipe Coating. National Bureau of Standards Corrosion Conference, 1937.
2. J. W. McBurney. Indentation of Asphalt Tile. *Proceedings of the American Society for Testing Materials,* 34, 591. Part 2, (1934) Discussion.

Reply by Lyle R. Sheppard:

Mr. Hadley asked two pertinent questions concerning the justification of the quality control test program. He answers his own first question, "does existing data indicate a need for such a check," by using experienced men as inspectors during mastic application. Several cases of unsatisfactory performance of mastics, particularly on the Eastern and Southern Coasts, have been noted. These possibly indicate that the present method of quality checking is inadequate. Future use and performance experience will tell whether the proposed method is better than the present. The four plants now applying asphalt mastic apparently think the proposals have some possibility of merit for they have all asked information concerning the tests and made arrangements so they can make them.

It is gratifying to note that the values of current requirements of Mr. Hadley's and those given in the paper correspond so closely. It is also gratifying to note that the volume of necessary binder determined by Mr. Hadley (20 percent) and that given in the paper (22 percent) are so close.

It is agreed with Mr. Hadley that the decrease in coating electrical resistance with time is the most important coating effect to measure. But it is necessary to know the initial coating characteristics for comparison with those same characteristics after the decrease in resistance. By such a method, improvements in long time electrical properties possibly can be made.

This company has other coatings with greater electrical resistance than the recently applied mastic. However, they are not always suitable under the same conditions as mastic. By study of the properties of these and mastic it is hoped that the mastic can be made as good in its environment as the others are in theirs.

As with Dr. Gardiner's comments (see Page 90) it should be realized that the wide variation in penetration data (Figure 1) could have been partially due to the variations in the binder characteristics for they were not constant.

Laboratory tests were made on finished mastics as they were received, and as reformed and pressured to 1500 psi. This was done because most mastic properties have very little change above 1500 psi forming pressure. A comparison of the two property values, where possible, gave an idea whether any particular property was attaining its possible optimum value or not.

Mr. Hadley favors weighted concrete over weighted mastic where buoyance is a problem on pipe lines. The advantages of mastic over concrete are flexibility, increased electrical resistance, and normally initial cost. Performance was not determined on weighted mastic.

Comments by William G. Creel, H. C. Price Co., Harvey, La.:

As the manufacturer of the asphalt mastic tested in the first program, we wish to state that we are in accord with Mr. Sheppard's paper, and believe that his report and his tests will contribute to the further improvement of Somastic quality. However, we would like to elaborate on several of his points.

Mr. Sheppard makes the statement, "That the proportioning of binder and aggregate was the operation least under control during the mastic operation." We are continually endeavoring to obtain still better control. One change we have made since his tests is the installation of a 1¼-inch asphalt spray bar at the rear of the mixer to better distribute the asphalt. Prior to this, the asphalt was fed into the rear of the mixer from the open end of a pipe.

Since the installation of the spray bar, the tests run on the mastic in question using Mr. Sheppard's method by independent laboratories have shown a range in asphalt content from 11.24 percent to 12.73 percent, a spread of only 1.5 percent.

For the benefit of readers who may not be familiar with our mixing facilities, I would like to give you a brief resume. The asphalt is fed from a preheater by a Kinney Rotary Liquid positive displacement pump direct to a spray bar at the rear of a Barber-Greene pugmill which is the mixing chamber. The sand passes from a sand dryer through a 5′ by 4′ vibrating screen into a hopper. At the bottom of this hopper is a vane wheel which meters the sand into the screw conveyor. The asbestos is metered by means of a special wound screw into the same conveyor. The limedust is metered by a drag chain into the same conveyor. An elevator then carries the material from the conveyor to a 10′ long ribbon screw which acts as a premixer. From the premixer the material is discharged into the pugmill, which is 4′ long, 3′ wide, and 2′ deep. All units with the exception of the pugmill are driven by one variable speed drive, so all metering units increase or decrease simultaneously. The material is moved forward in the mixer by twin paddle shafts. Although our maximum tonnage is approximately 30 tons an hour, this pugmill has a rated capacity of 70 tons an hour for bituminous material. The material remains in the mixer a minimum of 2.5 minutes. From the mixer it discharges into a 12″ diameter x 9′ long transverse screw conveyor which feeds the pump that applies the material to the pipe. In order to check our proportions, we frequently calibrate the plant by weighing the individual components in relation to the asphalt.

I have described the mixing procedure in detail to illustrate that we try to get the maximum control practical.

We agree with Mr. Sheppard's testing procedure. Although we believe his tests are accurate, we do not believe that any large quantity, even enough to coat a full joint of pipe, was produced with either the extremely high percentage of asphalt (19.56 percent) or the very low percentage of asphalt (8.34 percent), because we have found that the mastic in question will not adhere to the pipe with these percentages, and we cannot believe that with our present metering system it is possible. We are of the opinion that these percentages of asphalt content represented a small localized area on the pipe.

Mr. Sheppard found an average weight per cubic foot of 131.4 lbs. For several years we have known that we were wasting some of our larger sand particles in our reject sand. That is particles which would normally pass a 6 mesh screen, but be retained on an 8 or 10 mesh screen. This would account for a less dense coating material and, consequently, a lower weight per cubic foot. All our sand is rescreened prior to entering our mixing equipment.

In August we redesigned our screening facilities and installed a larger, more efficient screen. Since then our weight has averaged 134.6 lbs. with a low of 133.3 lbs. and a high of 137.28 lbs.

In designing the mix, we agree with Mr. Sheppard's statement that proportioning should be done by volume. However, once the design is completed, the proper proportion of each component should be converted to percentages by weight for reasons of practicality. The only practical method to check the actual material proportions in production is to check them by weight, actually weighing the individual components.

We wish to express our appreciation for the wonderful co-operation received from Mr. Sheppard and for the information and ideas he gave us.

Reply by Lyle R. Sheppard:

It is gratifying to note that our tests have already resulted in two improvements:

1. A spray bar has been added for the better introduction of the asphalt binder to the aggregate in the mixer. This has resulted in a closer control of the percentages between the binder and aggregate (1.5 percent by weight range of the binder).

2. A larger and more efficient aggregate screen has allowed the use of slightly larger sand particles, thus increasing the average bulk density from 131.4 to 134.6 lbs. per cu. ft.

It is unfortunate that the paper did not make it clear that percentage of binder by volume should be used only in designing the mastic mixture. Actual application proportioning can be done by either volume or weight depending upon the type of equipment used.

Comments by F. M. Matheny, Pipe Linings, Wilmington, Calif.:

I am in general agreement with Mr. Sheppard's report on physical control of asphalt mastic coating by the use of approved methods of detecting inferior coating at the source of application. However, I feel that regarding the formulation of the mastic, several factors were not brought out in his report, which in my opinion have a direct bearing on the production and the quality control of asphalt mastic coatings.

First, let us clarify a point in the method of proportioning the materials that make up the coating. In our method of calibrating (setting the feed devices to properly proportion the materials to the mixer), we use weight percentages. Our plant operates on volume metering: therefore, we convert volume to weight to calibrate. In other words, all of our operation of mixing and applying is by volume; our control settings are by volume, but our method of setting up is by weight, which is then converted to volume.

The second part of the control is the ingredients that comprise the mix. The asphalt, being a refined product, can be controlled. The rock dust, being an almost pure limestone and ground to specification, can be controlled. This leaves the asbestos fiber and sand as the two parts of the mix over which we have the least control. The fiber percentage is very small, but its quality is important to give strength to the coating. The sand being from 65 percent to 66 percent by weight of the total mix, reflects greatly on the quality of the finished product. Clean, sharp, hard and dense sand, properly graded to give the greatest volume density, with the least amount of binder, should give the best coating. But as sand is a natural product mined from pits, it is the most difficult of the ingredients to control. Only by a costly operation could a continuous supply of ideal sand be assured.

We have, for this reason, set up a graph giving upper and lower limits, and assume our material to be of such quality as to give a good coating if it falls within these limits. In general, this requires a clean, hard, nonmicaceous, well graded granular sand, 100 percent passing a No. 6 mesh, U. S. Standard screen, and 97 percent to 100 percent being retained on a No. 200 mesh, U. S. Standard screen (coatings 7/16" and over).

Sand has several other properties which, while not affecting the visual appearance or application materially, can affect the quality.

The thermal conductivity of sands varies. Some sands will absorb heat more rapidly than others; thus, each grain is more uniformly heated through, and the apparent temperature reading, as it comes from the heater, will be near the true temperature as it is mixed with the other ingredients. Thus, the application temperature will be near the mixing temperature. Sand grains, however, that have a low conductivity will take a surface heat which will show the correct apparent temperature. Yet, due to the slower absorption of heat, the center of the sand grains will continue to absorb heat after it has been introduced into the mix, thus lowering the temperature of the sand below the apparent temperature.

In dealing with the weight of the sand, we must differentiate between volumetric weight and molecular weight. Molecular weight would have very little effect on the mix, where volume metering of the ingredients is used. But where the weight variation is caused by porosity of the sand grains, it can have an effect on the proportions of binder to sand by absorbing the binder and leaving less to mix with the stone dust and fiber. The porosity of the sand grains can have another effect which, though minor, should be mentioned: the moisture content. Porosity of the sand grains reflects the amount of retained moisture in the sand after heating. This consists of the free moisture on the outside of the grains and that which can be driven out of the pores by expansion, caused by the changing of the water into steam. There is still that percentage of moisture left in the sand which was the steam at pressure equilibrium in the grains. It can be assumed that the more moisture contained in the sand, the longer it is necessary to hold in the heater, or the more elevated the heat must be to give a correct mixing temperature.

While each of the aforementioned, if taken independently, are small percentagewise, they could, and very likely do have considerable bearing on the quality of the finished product.

No mention in the report was given directly to primers. Adhesion was referred to as though the mastic was applied directly to the hot plates. Through our experiments it would seem that the adhesion of the mastic is as good to bare heated pipe as it is to primed pipe. It would probably be better if adequate control of the temperature of the pipe were maintained.

To summarize: the volumetric metering of the materials would seem to be the most practical way, from a production standpoint, to compound the mastic for coating. The lowering of the asphaltic content would perhaps be beneficial in some respects; however, I feel that in order to do this, the sand quality would have to be more rigidly controlled, which would add materially to the cost of the finished product.

Reply by Lyle R. Sheppard:

Mr. Matheny has noted the influence of application pipe temperature on the mastic adhesion. This was noted during the testing but not mentioned in the paper. Application to cold pipe adversely affects adhesion, causing a hard, brittle bond between the mastic and pipe. Conversely, mastic applied, even without primer, to pipe heated to approximately the temperature of the mastic mixture gives high values of adhesion. For this reason preheating of the pipe has been suggested to various applicators. Too rapid cooling of coated pipe can also cause similar adhesion effects as those encountered in application to cold pipe.

Comments by Earl W. Klinger:

Asphalt protective coatings for pipe lines is a field of particular interest to the asphalt industry and many reports have been published on this subject. However, there is considerable information which, for various reasons, has been distributed only within the individual companies. It is gratifying that Mr. Sheppard's company has given him permission to present the results of this work to the NACE Denver Conference.

Perhaps a brief review of events leading to publication of The Asphalt Institute Construction Specifications, "Asphalt Protective Coatings for Pipe Lines," Construction Series Number 96, first issued in May 1954, will serve to emphasize the importance of publication of data such as these reported by Mr. Sheppard for asphalt mastic coatings. Such data are essential in the preparation of adequate national specifications which will fully protect the interests of the customer, contractor and supplier.

One of the references given by Mr. Sheppard was the National Bureau of Standards Circular 450, issued in 1945, reporting results on their field burial program which was initiated in 1922. Coating materials were furnished by the various suppliers and serious efforts were made to include all known materials used for pipe line coatings. Very briefly stated, Circular 450 showed a definite relationship between performance and thickness, density, hardness and flexibility of coating to mention only a few of the characteristics studied. To bring this reference up to date, Circular 450 has been superseded by the National Bureau of Standards Circular 579 issued April 1957.

Beginning about 1938, The Asphalt Institute issued several publications written in the form of recommended practice for asphalt coatings on metal surfaces. Following the release of Circular 450, it was believed desirable to write national specifications for materials and construction procedures for asphalt pipe line coatings and a study was made on the availability of pertinent data. It was found that in order to write adequate national specifications, it would be necessary to find some means to obtain the release of unpublished data held by the individual companies. Further discussions lead to the formation of a Project Committee on Asphalt Pipe Line Coatings charged with preparing the required specifications and full cooperation of the Member Companies of The Asphalt Institute on release of essential information for the specific use of the committee members was obtained. The publication of Construction Series Number 96 issued in May 1954 and referred to above resulted from this wholehearted cooperation.

Mention might be made also of NACE Technical Unit Committee T-2H and the subsequent revision of Construction Series Number 96 in December 1958 to conform essentially to NACE Publications 57-11, 57-14 and 58-12. The experience of the members of NACE T-2H was invaluable in further resolving the sectional differences in practice as reported above.

The data reported by Mr. Sheppard together with published data for other types of asphalt pipe line coating, if available at that time, would have been of considerable assistance in our effort to write adequate national specifications. Some discussion as to the need for strength tests in addition to the Tensile Strength of the mastic coating lead to the rather indefinie conclusion that the Tensile Strength Test was the only one available with the required correlation with field performance. Mr. Sheppard has reported that the Blunt Needle Penetration Test as developed by Shell Pipe Line Corporation is satisfactory for both design and control of asphalt mastic coatings. Further study and evaluation of this penetration test and perhaps other types of strength or stability tests offer hope of future improvement in national specifications for asphalt mastic coatings.

Mr. Sheppard in his Summary lists a number of factors which require further study and it is hoped that Shell Pipe Line Corporation will continue this work and publish their findings. We should also hope to see more published reports by others thereby providing data which would assist to improve our national specifications for asphalt pipe line coatings.

Reply by Lyle R. Sheppard:

On page 177 of the Bureau of Standards Circular C 450, the effects on coating performance of thickness, density, hardness and flexibility are given as found by those investigators. It was mentioned that rigidity and flexibility in a coating were difficult to reconcile. Possibly if the reasons for having these characteristics in a pipe line coating are given, it would be easier to solve the problem of combining these apparently conflicting characteristics in a single coating. Rigidity is required in a coating to resist the damage done by the weight of pipe, soil and product on the coating. Flexibility is required in order to resist coating deformation by the pipe because of pressure or temperature changes.

If changes in what we chose to call the characteristics of plasticity is used in the coating, the necessary rigidity and flexibility cannot be attained simultaneously. While "plasticity" may not be the proper term, we chose to give it the meaning of continued movement under applied stress with very little or no tendency to return to the original position when the stress is removed (like putty). A coating with this characteristic, if made more rigid becomes less flexible and vice versa.

If, however, the characteristic of elasticity is changed, the necessary rigidity and flexibility can be attained simultaneously. Again the wrong meaning of elasticity may be used. We mean by elasticity the ability of a material to move under applied stress and to return to its original position after stress is removed (like rubber). Thus we believe that elasticity, as defined, should be incorporated into pipe line coatings.

We agree with Mr. Klinger that more publicity should be given to individual coating findings and testing techniques.

Comments by H. C. Knickerbocker, New Orleans, La.:

Mr. Sheppard's paper indicates that asphalt mastic coating has proved for years to be an excellent corrosion protection and that it is now recognized in its newer function in which it is used as a weight factor to produce a required negative buoyancy.

The programs represent a study of both types of coating and, while there has been no direct attempt at comparison, a partial comparison is inevitable. Had Mr. Sheppard intended a comparison, he would have followed identical procedures in sampling and testing of both coatings. It is regrettable that he did not have an equal opportunity in the second program for sampling and testing as in the first. Because he did not have that opportunity, some explanation and clarification may help in a proper evaluation of each type. This is an attempt at that explanation and clarification.

Actually, very little need be said about the coating studied in the first program, as it is well known, widely used and universally acknowledged to give excellent corrosion protection. The one fact to be noted, however, is that when weight is required, that weight is provided by other materials and methods. Usually this is accomplished by a concrete jacket applied over the corrosion protective coat. Although concrete has been used for this purpose on offshore pipe lines for a number of years, it has not proved universally satisfactory to the transmission line owners. One of the principal objections has been the difficulty in getting it into the water without cracking, due to overbend or wave action, as it is laid off of the barge.

The mastic in the second program, as indicated earlier, is designed to serve the dual purpose of providing adequate corrosion protection plus necessary weight. It is, of course, obvious that if asphalt mastic gives corrosion protection, this should be more than adequate for that purpose, since in order to provide weight, it is first thickened. Further weight is obtained by the design of formulations carrying varying amounts of heavy aggregate, as needed.

The design of mastic formulations for producing weight is not a simple matter. Neither is it a simple matter to control the mix after it is designed.

Some of the controlling factors (but by no means all) are: the gradation of the raw aggregates, the weights of these ingredients and ultimately the bulk density, the penetration and softening points of the binder, the extrudability of the mix, the heat, and last, but by no means least important, economy.

Mr. Sheppard has mentioned the inclusion of rubber in the mastic of the second program. The rubber in the mix to some extent limits the penetration and softening point of the binder that may be used. That is, to use a lower penetration and higher softening point asphalt, some additional heat would be required. This could be injurious to the rubber in the mix.

At this time it would seem appropriate to refer to Table 3 of Mr. Sheppard's paper. Here it will be noted that the four samples were taken on 3/57, 8/57, 1/58 and 2/59. The first three of these samples were from coating applied by a barge mounted coating plant. The fourth sample came from coating applied by a new and modern land based plant. The mix formulations in the first three samples were materially different from that in the fourth. The mixes on the barge-lay coatings were based on the use of emulsified asphalt, and powdered asphalt as binder material. The base asphalt in the emulsion was about 70 penetration. The penetration was reduced to 30 to 35 by the addition of powdered asphalt. This was considered low enough as the coated pipe was going directly into the water. Whether or not this high penetration was good or bad practice is now a moot question, since the barge coating method is obsolete and is no longer used.

The fourth sample dated 2/59 was from pipe coated in the land-based plant, but is still not quite representative of the most recent practice. At the time this sample was taken, the binder was purchased under a specification which required (among other things) a penetration between 20 and 30 and a softening point between 160 and 170. About June 1959 the limits on penetration were reduced to 20 to 22 and the softening point raised to 168 to 170. These changes brought the asphalt binder closer to the characteristics in the NACE specifications for Grade II. Additional tests are being made to permit, if possible, further approach to Grade II specifications without injuring the rubber.

On the conclusions drawn from both programs the following comments are offered:

1. The blunt needle test is a good quality control.

2. The control of proportioning of binder and aggregate in the land based plant now used in the second program should be very accurate as all ingredients are weighed on automatic scales in a weigh-batch mixing plant of most modern design.

3. No test of volumetric proportioning could be made easily on the Program 2 mastic as all plant design was for weight proportioning.

Nos. 4, 5, 6 and 7 are conclusions that cannot be denied.

8. The conclusion here must be agreed to, but it would seem proper to add that some method of determining the pressure of the mix, as it passes through the extrusion die on the production plant should be developed.

Reply by Lyle R. Sheppard:

Mr. Knickerbocker has had considerable experience with applying asphalt mastic for weight as well as corrosion protection. Weight is obtained by both increasing the coating thickness and the bulk density of the aggregate. This additional hot weighted mastic is held on the pipe by a spirally wound fabric until the mastic has been cooled to its normal hardness.

The addition of rubber to the binder has allowed lower softening point asphalts, and thus lower application extrusion temperature, to be used. At the same time elasticity has been added to the finished mastic. The rubber addition increased the test specimen softening points approximately 5 degrees F and also increased the penetration or decreased the hardness. However, application temperature must be kept low in order not to damage the rubber.

It is hoped that someone will shortly develop an accurate method of measuring the extrusion pressure, as it is undoubtedly a factor in obtaining a more dense coating with the same proportioning.

Comments by E. W. Gardiner, California Research Corporation, San Francisco, Calif.:

The specifications issued a few years ago by the National Association of Corrosion Engineers and the Asphalt Institute were the result of several years of study by committees of these two institutions. They were based on the collected data and test methods in use by manufacturers and users of pipe coatings at that time. Mr. Sheppard does not say why he chose to use other procedures than those specified by the NACE for the evaluation of mastic coatings. I refer particularly to tests for adhesion (bond strength of primer), cohesion (tensile strength of mastic) and penetration (depression test). We do not know whether Mr. Sheppard's test procedures for adhesion and cohesion are better or poorer than those for bond strength of primer and tensile strength of mastic described in the NACE specifications. If Mr. Sheppard has data comparing the two sets of methods or has reason to believe his methods are superior, we suggest that he present his data to an appropriate committee of NACE, AI, or ASTM for study.

The use of a needle, even blunted, for determination of consistency of a mastic is of questionable value, because the mastic contains particles of aggregate larger in cross section than the effective diameter of the needle. In Figure 1 a 5-fold variation in penetration is reported for the same binder content and a 2-fold variation in binder content for the same penetration. We think that these variations may be the result of interference by the aggregate and certainly do not represent variations in quality of the mastic or lack of uniformity of manufacturing procedures. On the other hand, the depression test using a three-quarter inch diameter rod and fifty pounds per square inch pressure would not show nearly as much variation from sample to sample and adequately evaluates the ability of the mastic to resist clod pressure, a quality for which mastic coating is superior.

The customary procedure for recovery and determination of asphalt in materials of construction is ASTM D762-49. It is a modification of the original Abson procedure and gives fairly accurate results when all steps are followed carefully and, in addition, provides sufficient sample for further testing of the asphalt. We wonder why Mr. Sheppard chose to determine the percentage of binder by difference from percentage of ash using a method designed for determination of ash in coal and coke (ASTM D271), materials vastly different in mineral content from mastic pipe coating.

The values for percentage of binder reported by Mr. Sheppard in Table 2 show a variation that is inconsistent with practice. In our experience the operation of a blending plant for mastic pipe coating is so well controlled that a variation from 8.34 percent binder to 19.56 percent binder would be impossible. In fact, mastics containing these extremes of binder content could not be applied satisfactorily to pipe. At very low concentrations of binder the mastic would be so stiff that it probably could not be extruded and would be extremely difficult to compact on the pipe. At such high concentrations of binder as 19.56 percent, the mastic would be so soft after extrusion that it would slough off the pipe before cooling. We have analyzed many samples of mastic pipe coating for percentage of asphalt binder by the ASTM D762 method and find that the variation is well within the experimental error of the method and within the range of the specification. We suggest that in the future users of mastic pipe coating use ASTM D762 for determination of the asphalt binder in mastic pipe coating.

Mr. Sheppard arrived at several conclusions as the result of his work. He concluded that "proportioning of binder and aggregate was the operation least under control during mastic production." As discussed above, we cannot agree with this conclusion. Operators must and do control the proportions of their mastic blends most accurately in order to produce uniform products. The excellent service obtained from mastic-type coatings over thousands of miles of all sizes of pipe under all kinds of conditions is proof of the uniform quality of the product.

Mr. Sheppard also concluded that proportioning "should be done by volume rather than weight, regardless of the bulk density of the mastic." The specifications for weight percent of binder are based on using an aggregate of a fixed type as defined in the specification. Obviously, if the density of the aggregate is changed markedly, the percentage by weight of binder must be changed so that the resulting mastic will have the proper handling properties and adequate tensile strength, impact resistance, bending strength, resistance to deformation, and electrical characteristics. It may be easier for the manufacturer to operate his mixer by weight than by volume, or vice versa, but in either case the conversion from volume to weight should be direct when the density of the aggregate is known.

The conclusion that further work on extrusion speed, application temperature, and cooling after application is necessary does not agree with our experience. If such work were to result in more uniform coatings, giving still better protection, we would of course agree. However, the proof of better quality cannot be evaluated by laboratory tests, but requires extensive and expensive field tests covering many miles of pipe and many years of service. We doubt that such a program is warranted in view of the superior results now being obtained with mastic pipe coatings.

Reply by Lyle R. Sheppard:

It is very gratifying to have this paper criticized by Dr. Gardiner because of his vast experience in helping to formulate the present-day successful asphalt mastics. These criticisms have and are being very carefully considered.

Most of Dr. Gardiner's questions were about the testing techniques. It would be well to answer his questions in order:

1. **Adhesion.** Adhesion testing is done in the same manner as that specified by the Asphalt Institute with two exceptions. First, a test specimen 3 inches in diameter and 7 sq. in. in area is used instead of one 1.128 inch in diameter and 1 sq. in. in area. And secondly, the prepared specimen is allowed to cool to ambient temperature (approximately 77 F) at least sixteen hours instead of one hour before it is brought to test temperature in a water bath.

The reasons for making these changes in the test are:

a. The ratio of the cross-sectional area of the specimen to its circumference is increased 2.66 times by using the larger specimen. This type of tensional test is adversely affected by the "edge" effect. Thus measurement of this surface characteristic of the mastic has been made more accurate by enlarging the specimen.

b. The surface characteristic measured is an average of the breaking points of the small areas composing the entire surface. The larger specimen averages the breaking points on a larger area (7 times as great as the smaller area). This tends toward greater measurement accuracy.

c. Longer cooling allows time for possible internal stressing to come to an equilibrium, thus making more uniform and reproducible test results.

2. **Cohesion.** It is believed that the cohesion or tensile test made in this paper is basically more accurate and reproducible than that given by the Asphalt Institute (modified A.S.T.M.-C190—Tensile Strength of Cement Mortars). Following are reasons for believing this:

a. Asphalt Institute tests are made on 1 inch square specimens. In addition to the adverse "edge" effects mentioned under adhesion testing, an adverse "corner" effect is introduced where stressing at the four corners of the specimen is not uniform with that along the side edges. Enlarging the specimen and making it have a circular cross-sectional area gives similar advantages as those mentioned on adhesion testing.

b. Having the specimen sizes the same as those for adhesion testing allows direct comparison of the mastic properties of adhesion and cohesion under similar testing conditions.

3. **Blunt Needle Penetration.** Blunt needle penetration tests of finished mastic were used for two reasons mainly. First, it required minutes instead of hours (¾ inch diameter blunt rod) to make the test, and second, it was a modification of an ASTM test (D5—Needle Penetration of Bituminous Materials). Because of the probability of the small needle slipping between large aggregate particles or being stopped by them, it was found that averages of six individual tests could be repeated on the same mastic within ASTM tolerances for normal penetrations. This occurred even though no two of the twelve individual readings would be identical. This test had to be quick in order to be effective because it was used as a quality control test during application. The only time when this test, used for quality control, did not indicate application difficulty was when the added fibers were not willowed as they should have been.

Dr. Gardiner did not realize, in criticizing Figure 1 of the test results, that the binder softening point varied 15.5 F and was very definitely reflected in each of the points shown in this figure. If the binder had been quite uniform, his criticism would have been justified.

4. **Percentage of Binder.** Ashing tests simultaneously of both the finished mastic and the total dry aggregate were used to determine the proportioning of the binder and aggregate in the finished mastic. This is an indirect rather than a direct method but was used because it requires less time and attention than the direct method (ASTM-D762-49). It could not be applied when the dry aggregate was not available, such as when only the sample of finished mastic was available. In that case the direct extraction method would be used.

Actually, a modified ASTM-D762-49 method was used in a number of cases to verify the ashing method. The extraction method was modified, using a vacuum oven, so that the solvent was extracted without allowing the asphalt ever to obtain a temperature of more than 265 F instead of the 300 F plus used in the regular extraction test. It was found that by using this modified method an asphalt of known softening point and penetration could be extracted without changing these characteristics.

Comments by Gordon N. Scott, Los Angeles, California:

Mr. Sheppard is to be congratulated on his work to better the performance of asphalt mastic pipe coatings underground by providing additional methods for quality control at the plant. It would profit little to take great care in the field installation if the coating arrived defective at the site of laying.

The writer has long been interested in the performance of asphalt-mastic coating in the ground[1] but has been less concerned with manufacture. However, examinations of the coating in service have disclosed, among other things, breaks or discontinuities in the coating attributable to faulty manufacture.[2] These include unwillowed asbestos, particles of charred bitumen, and too high an asphalt content in the completed product. These are faults to the elimination of which Mr. Sheppard's efforts have been directed.

Quality control at the plant has long been in effect, as has been pointed out by other commentators. Asphalt is purchased on rigid specifications,[3] sand is carefully selected and the grading of it done with added lime for maximum density; "jeeping" of every joint as it emerges from the extrusion machine locates for repair the smallest holidays. The proper handling of the coated pipe in the plant and the loading of it on trucks adequately padded prevent abrasion; the use of modern construction methods and facilities insure as nearly as possible an unbroken coating in the backfilled ditch.

Some pipe line companies have included in the pipe-laying contract a "coating discontinuity survey" clause[1] which states that the company at its option may run a coating discontinuity survey over the pipeline one or two years following construction and hold the contractor accountable for any discontinuities which are found to be clearly attributable to errors of construction.

It has been clearly demonstrated[1, 2] that in a carefully constructed asphalt-mastic coated pipeline it is possible to obtain a coating essentially free of discontinuities. It has been suggested that a stage had been reached in both manufacture and construction where attention could be transferred from maintaining the pipe to maintaining the coating. The suggestion was made even though in most instances cathodic protection as a supplement to the coating was needed only on an exceedingly small scale. It seemed better to repair the coating than to risk damage to it by the current, which is usually most inefficiently applied, and to avoid the maintenance of a cathodic protection system, however small.

Cathodic Protecion Effectiveness

There is no question of the effectiveness of cathodic protection in corrosion prevention. Poorly coated pipelines, corroded to a point where operation is no longer economically feasible, may be continued in use with the aid of cathodic protection. Such cathodic protection, however, may be applied in such a way that the efficiency is very low. The consequences of imposing current on neighboring or crossing pipelines may in these cases become serious. The current, utilizing a large segment of earth in its passage from anode to pipeline, more or less closes the area to certain classes of pipe, including cast-iron, concrete cylinder pipe and the like, unless modified by high resistance coatings. Thus, whatever the justification for such usurpation, there is, in excessive cathodic protection, an element of injustice in the exclusion of these pipe from areas where high cathodic interference may develop.

The enormous benefits of cathodic protection are accompanied with certain other disadvantages of which probably the most serious is the destruction of the coating by the current. This problem has not yet received sufficient attention.

The transfer of current from (or to) an electrolytic medium to (or from) a metallic one is accompanied by chemical changes in proportion to the strength and duration of the current. If the soil is acid, that is, if the pH is less than, say 7, then the hydrogen ion under the influence of the current migrates through a flaw in the coating to be neutralized by lime, if used as a filler, or to be evolved as hydrogen gas on the metal surface. For this to happen it is necessary that the damaged coating be wetted so that electrical contact is established between soil and pipe.

The hydrogen ion is rarely in the form of a single, positively-charged atom, but is almost invariably combined with a molecule of water to form hydronium ion. So great is the affinity of hydrogen ion for water that the dissociation constant is an unbelievably small number. The charged atom, H^+, practically does not exist *per se*.[4] Thus, according to the equation

$$H_2O \cdot H^+ \text{ or } H_3O^+ = H^+ + H_2O, \quad K = 1 \times 10^{-130} \qquad (1)$$

As hydrogen ion migrates to the cathodically protected surface, one would expect it to be accompanied by a molecule of water which would remain in the abrasion or at the metal surface when the hydrogen ion was neutralized by the lime or evolved as hydrogen gas,

$$2H_3O^+ + 2e^- = H_2 + 2H_2O \qquad (2)$$

In acid soils, therefore, water would be expected beneath electrolytically conducting coatings under cathodic protection. In alkaline soils other positive ions such as sodium, potassium, calcium and magnesium, which also migrate to the metal surface, will form hydroxides as hydrogen is evolved from water:[2]

$$2\,H_2O + 2\,Na^+ + 2e^- = 2\text{ Na (OH)} + H_2 \qquad (3)$$

Thus, which of the indicated reactions will take place depends in part upon the pH and upon the moisture conditions in the soil. If the soil is acid it may be confidently expected that water will proceed through a conducting coating and be deposited at the metal surface eventually to form hydroxides in ever increasing concentration as the current flows, even though these negatively charged hydroxyl ions are repelled by the like charge on the cathode. Concurrently positive metallic ions accumulate about the abraded coating surface to form hydroxides which are converted in time to carbonates to cement the soil about the damaged coating area, as recorded in the photographs in Miltner.[2]

High concentrations of sodium hydroxide will saponify certain bituminous materials and thus impair the bond to metal. Excessive saponification results in the ultimate destruction of the pipe coating as such. Concentrated sodium hydroxide reacts with materials like asbestos, sand and glass. Boron glasses are more resistant to alkali than are soda glasses.

Certain mechanical effects result from the alkalinity and hydrogen gas produced in cathodic protection. Loosening of the bond of coating to pipe has just been mentioned. If the current density is high, blistering of the coating may occur as a consequence of the rapid liberation of hydrogen gas.

Rusting of pipe happens under some conditions. If the concentration of hydroxide ion is high and there is an abundance of oxygen in a moist, open soil, corrosion can occur even if the pipe is under cathodic protection. This effect, forecast in a measure by Pourbaix,[5] has recently been observed in the field by Paisley and Scott.[6]

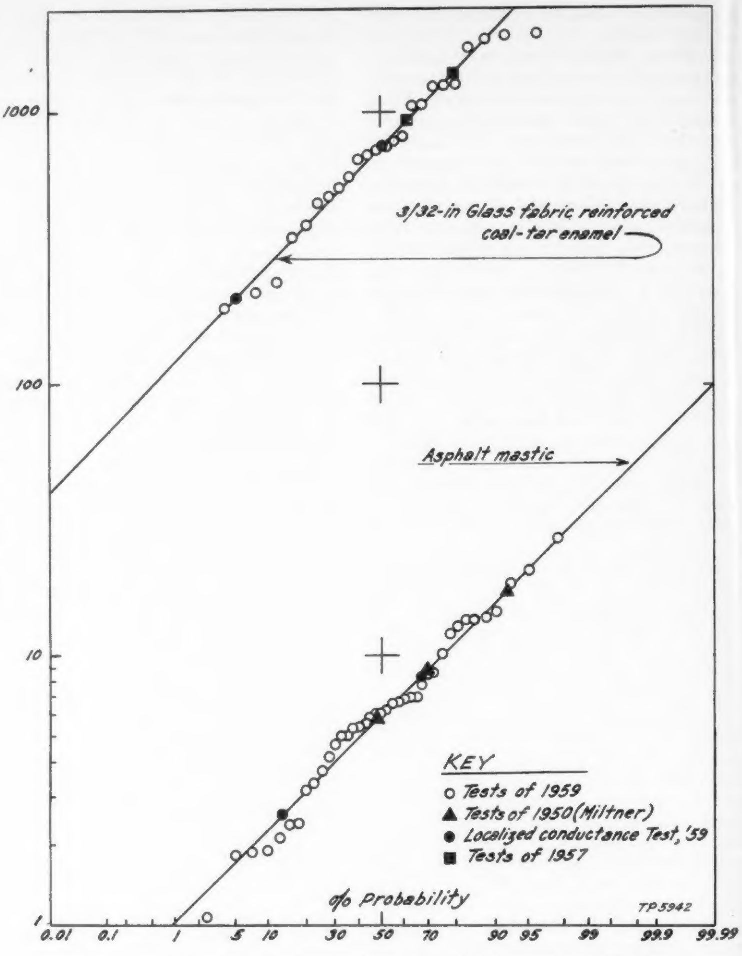
A consequence of these several undesirable effects of the current on the coating materials is a loss in the insulating property of the coating. The change in this property, which may be measured as the coating conductance, permits further increase in current density at the defects or damaged areas so that soon the controlling protective potential remote from the point-of-drainage is lost. To restore the remote potential to a protective level requires an increase in applied current or intermediate cathodic protection stations. It follows, therefore, that cathodic protection should be accomplished sparingly, most efficiently, and with a minimum attentuation of current, (that is, with a maximum economical number of cathodic protection stations consistent with maintenance costs and a long life of coating which should be as free of holidays as possible, within reasonable economic bounds).

Case Histories

These arguments can be made more convincing by describing specific cases, which are not necessarily representative of coatings of like construction.

Case 1.

In 1947, Gas Industrial de Monterrey built a coated, 14-inch diameter, ¼-inch wall pipeline from Reynosa to Monterrey, N. L., Mexico, a distance of 130 miles. Shortly after construction, on competent corrosion engineering advice, three gas-engine driven generators were installed, to provide the pipeline with further corrosion protection. No power was available anywhere except at the extremities of the line. In 1951, after a potential survey over the line, an additional generator unit was installed. The following year two more gas driven units were located at intermediate points. Rectifiers were added at the same time at Reynosa and at Monterrey. This and subsequent history of the installation of the presently operating 14 cathodic protecting stations is shown in Discussion Table 1.



Discussion Figure 1—Coating conductance conforms to logarithmic normal probability law.

DISCUSSION TABLE 1—Chronological Order of Installation of Cathodic Protection Units (Current Outputs Are As of December 1959)

Year	Age of System (Years)	Cathodic Protection Units	Total Units	Current Output, Amps.
1947	0	Pipe Line Built	0	. . .
1948	1	48*	1	30
		129	2	65
		180	3	40
1951	4	92	4	100
1952	5	75	5	50
		147	6	40
		0 Reynosa (Rectifier)	7	6
		210 Monterrey (Rectifier)	8	24
1953	6	32	9	35
		110	10	50
1955	8	40	11	21
		84	12	40
1956	9	165	13	50
1958	11	22	14	12
		Total	14 C.P. Sta.	568 Amps.

* The numbers are the approximate distances in kilometers of the units measured from Monterrey towards Reynosa.

The unit conductance of a pipeline coating measures well the performance of the coating. Kulman,[7] applying a probability technique described by the writer[8, 9, 10] has shown that the probability distribution of coating conductance follows the logarithmic normal probability law. Accordingly, one may plot coating conductances on a logarithmic probability chart as in Discussion Figure 1. The circles, defining the upper curve, represent coating conductances measured in January 1959 by engineers of the gas company and by the writer over approximately 25 miles of the 14-inch line between Km 21 and 58. These conductances have been reduced from a per running meter to a "per square foot of pipe surface" basis and a conductance which would be expected in a 1,000 ohm-cm. soil.

To determine whether the proximity

of the anode to the pipeline was an influencing factor in damage to coating by cathodic protection approximately 15 associated line current and potential measurements were made in 1957 both upstream and downstream from the point of drainage at Km 48 over a distance of about 3,000 feet each way.

In attenuation theory, the pipe voltage is referred to remote earth so that if the coating is poor in insulating quality a significant fraction of the voltage drop from remote earth may be through the soil and the voltage customarily measured on the surface over the pipe will be too low in value. If the soil resistivity and coating conductance were both uniform, it would be simple to calculate the coating conductance from the voltage alone. Current measurements are not subject to this difficulty, and so it is possible to calculate the coating conductance accurately from measurements of the pipe gradient without knowing the pipe voltage with respect to soil. The average conductances calculated from the gradients for both the upstream and downstream measurements, corrected to a per square foot basis in a 1000 ohm-cm soil, are located as black squares at the appropriate conductance values on the probability curve.

Further to test the coating, several excavations were made in 1959 up to more than $\frac{1}{2}$ mile distances from the point-of-drainage at Km 147 where the soil conductivity, in the order of 10,000 micromho per cm, is exceedingly high. This localized conductance test was made using a cotton outing flannel pad and a wire screen wrapped around the coated pipe as described in Circular 579.[11] The coating in this area had never been subjected to as much as 1.5 volts to the copper sulfate electrode. Nevertheless, it was found impossible to measure the conductance of the coating or even the coating thickness at points close to the generator since the coating on excavation either came off the bottom of the pipe with the soil clods or the coating was so badly blistered or loosened that the application of the conductance pad could not be made without damaging the fragile coating and thus invalidating the test. Nevertheless, two measurements could be made on reasonably unbroken coating. The reduced conductances are plotted as black dots on the upper curve of the figure. It is somewhat astonishing that despite the poor condition of the coating these corrected measurements fall at or below the mean conductivity.

The 24 tests of 1959 shown as clear circles conform well to the logarithmic probability law. The several different kinds of test apparently give results which are comparable. The supplementary measurements fall within the range of the 24 measurements of 1959.

The area through which the pipeline passes is arid. The soils are, accordingly, alkaline. At Km 147 the high conductivity (resistivity of 100 ohm-cm and less) indicates an exceedingly high salt content in the soil. Although the protective potential has never been excessive here, the current density must certainly be high since the rate of hydrogen production at the pipe surface has been sufficiently rapid to cause blistering of the coating, solution of the glass reinforcing fabric and loosening of the bond. Although the pH at the pipe surface was neither measured directly nor estimated from the polarization potential, it was sufficiently high to dissolve the lead out of the solder used to connect the cathode cable to the pipe. Although a small piece of glass fabric was found protruding out of the coating and into the soil at one location, no fabric could be found in the coating elsewhere except at the most distant point (0.62 miles) downstream from the generator (operating at 40 amperes output). At that location one small piece of coating occasionally would remain attached to a second piece by a fiber of glass.

Case No. 2

Similar coating conductance measurements have been made by Paisley and Scott[6] over the 335 mile long $6\frac{5}{8}$ inch diameter, asphalt-mastic coated pipe described by Miltner. This line was laid in 1941 and has been under cathodic protection since date of laying with four rectifiers near major river crossings and a lateral in an industrial area. The protection was completed with a fifth rectifier installed at the north end of the line about 1951.

The 39 conductances plotted as circles in the lower part of Discussion Figure 1 were measured in August and September 1959. Two localized tests at randomly selected locations in the bottom lands of the Missouri River yielded the reduced conductances plotted as dots on the curve. The three unit coating conductance measurements reported by Miltner have been reduced to standard soil conditions and are shown as black triangles on the probability curve. As in Case 1, the three sets of measurements fall within the range of conductances determined by the 1959 measurements, which conform to the logarithmic normal probability law. Progressive deterioration of this coating as a whole is not evident.

Not all of the coating discontinuities reported by Miltner were excavated for inspection in 1950. To gain an idea if the current had further affected the coating, as had been concluded by Miltner, two conveniently located coating discontinuities were excavated and examined. At both locations the discontinuities appeared to have been enlarged in area by the destructive action of the chemical products formed at the pipe surface by the cathodic protection current. There was evidence of disintegration of the coating material. At the one location (Mile Post 158.16) only a thin layer of surface coating remained of the original $\frac{1}{2}$-inch thick coating. It appears that saponification of the asphalt binder occurred. At the second location (Mile Post 189.79) it was found that the discontinuity now exceeded the length of the 4 foot bell-hole and it is certain, therefore, that this discontinuity would have been classified as a major break in the 1950 survey and therefore would have been opened for inspection then. As at the other excavation the indication is that the coating is progressively disintegrating at the holidays.

A most surprising feature at both locations was that the pipe was rusted to a considerable extent, although no pitting of consequence was found in the metal. At both locations the soil was reasonably dry so that the moisture film over the pipe surface must have been in contact with atmospheric oxygen at all times. Traces of rust at some discontinuities were reported by Miltner but he supposed that the rusting occurred after the coating was removed and the pipe exposed to air.

Hydroxides Produced in Cathodic Protection

It has long been known that cathodic protection must be applied to lead in moderation since lead is soluble in the hydroxide ion produced at the metal surface. Zinc and aluminum are other metals in addition to iron and lead which form amphoteric hydroxides and must therefore only be put under mild cathodic protection to prevent corrosion by the hydroxides produced by the current.

The cathodic corrosion of lead was illustrated on the 14-inch line where the copper cathodic protection cable was soldered onto the pipe. The current density is at its highest at the point-of-drainage and accordingly the lead in the solder dissolved in the concentrated alkali produced there by the current. The solution of the lead is believed to have so increased the resistance of one soldered connection that arcing occurred, which resulted in puncturing the pipe to cause a leak.

Iron in the presence of strong alkali may behave in a similar way. Passivation of iron occurs generally in the pH range 8.5 to 12.5 in the absence of anions like chloride and sulfate. However, if the current density is such that the pH exceeds this limit, the passivating film will be dissolved by the hydroxide ion and, under the potential afforded by oxygen, iron will dissolve to form soluble ferrites or ferrates which ultimately precipitate as hydrated ferric oxide.

Evidently there is much chemistry to be considered in cathodic protection. It is a consequence thereof that, consistent with other features, an insulating coating should have as high an electrical resistance as possible, that it be laid in an unbroken condition, and that cathodic protection be used efficiently and sparingly to keep the current to a minimum.

To insure that the plant coating has a maximum electrical resistance is Sheppard's objective. That the laid coating retain as much as possible of this quality is a joint responsibility of the company and the laying contractor. But the long time performance of the coating is in the hands of the corrosion engineer.

There are many reasons why cathodic protection is an extraordinarily inefficient process. There are but two of these reasons which the writer wishes to discuss since these bear directly upon Sheppard's objective and the work of the corrosion engineer. Sheppard's objective hardly seems warranted if the coating is subse-

DISCUSSION TABLE 2—Anodic Potentials With Respect to Reference Half Cells

Salt	Standard Hydrogen, Volts	Saturated Calomel, Volts	Saturated Copper Sulfate, Volts
$FeCl_2.2H_20$	—0.405	—.647	—.705
$FeCl_2.4H_20$	—0.410	—.652	—.710
$FeS0_4.7H_20$	—0.468	—.710	—.768

quently to be abused to possible disruption by excessive and inefficient cathodic protection.

The two problems of immediate interest are: (1) the minimum protective potential of the pipe with respect to earth (since this potential is an important factor in the performance of the coating because everywhere toward the point of drainage other than at the remote point of minimum potential the applied current is in ever increasing excess over requirements), and (2) the maximum permissible alkalinity at the pipe surface to avoid damage to the components of a composite pipe coating. The writer can forsee that the corrosion engineer will in the distant future base his design of cathodic protection in the narrow range, fixed by his particular environment determined from a soil corrosion survey, of a minimum protective potential and a maximum allowable polarization potential at the point of drainage as fixed by the solubility in alkali of either iron or components of the composite coating. The argument can obviously be extended to other metals such as aluminum, lead and zinc but the criteria will in each instance be different as will be seen in the exposition to follow.

There are divergent opinions on a minimum protective potential for cathodic protection. Under the most severe conditions of underground corrosion, a minimum potential of minus 0.850 volts[12] with respect to the copper sulfate electrode in adjacent earth has been generally accepted as a practical criterion. Schwerdtfeger[13, 14, 15] has consistently found in laboratory experiments optimum protection at 0.77 volts to the saturated calomel electrode which voltage corresponds roughly to the generally accepted practical criterion quoted above.

But there are several other approaches to the problem, which all lead to desirably lower minimum protective potentials. One such approach will be discussed. If we accept for purposes of illustration the conclusion of Mears and Brown[14] that complete cathodic protection is attained only when the metal is cathodically polarized to the open-circuit potential of the most anodic area on the metal's surface, we can establish from electrochemical data a minimum cathodic polarization which will just barely protect the metal in a moist (not saturated) soil where the formation of crystals of hydrated $FeSO_4$ or $FeCl_2$ is incipient.

These anodic potentials in volts with respect to the several reference half-cells are shown in Discussion Table 2.

The conversions from the standard hydrogen electrode were made from the value of 0.242 volts for the saturated calomel electrode and 0.300 volts for the saturated copper sulphate electrode. The latter potential was estimated by the writer[16] from activity coefficients and the standard electrode potential of the half-cell. This value is in excellent agreement with the result of more sophisticated calculations of deBethune[17] et al. of 0.298 volt.

The calculation of the potential of the iron sulfate half-cell follows exactly the procedure illustrated by deBethune, but, in the absence of published free energy data for the chlorides, the potentials were calculated from heats of formation and estimates of the entropies of the hydrated salts.

Such potentials as citeria of protection, below which it would be imprudent to go, find support in practice. Protection of the Reynosa-Monterrey pipeline, which has been complete except as earlier noted, is based upon a minimum pipe potential of 0.800 volt to the copper sulfate electrode positioned over the pipe. Even this potential is not actually met over all of the pipeline at any time. The lowest potential found on a recent survey was 0.72 volt.

The writer is aware of failure of a minimum potential of 0.850 volt to stop leaks on old pipe and is advised of the necessity for a full volt in an instance. On the other hand he has many leak-free pipelines with minimum protective potentials ranging from 0.700 volt. Dow,[18] content with 0.800 volt for complete protection, considers the range from 0.700 to 0.800 as substantially protective.

It has been shown[16] that a cathodically protected pipeline is essentially a hydrogen electrode with a ceiling potential of about 1.22 volts. More recent tests confirm that the field-measured polarization potential serves to measure the pH at the metal surface and conversely that pH measurements at the metal surface foretell the polarization potential, all with reasonable accuracy.

Thus, from this fact and the relation[16]

$$E = 0.300 + 0.05916 \text{ pH}$$

it is evident that, at the minimum design criterion of 0.850 volt, the pH will be about 9.3. This is a minimum pH so that everywhere else and increasingly toward the point-of-drainage the pH will be higher. The question then is what maximum pH is permissible at the point of drainage to avoid damage to coating materials. If they cannot withstand an alkalinity greater than .01 Normal (pH 12) then the polarization voltage at the point of drainage should not be numerically greater than 1.01 volts with respect to the copper sulfate electrode in adjacent earth. According to Pourbaix, iron is unstable at a pH greater than 12.5 corresponding to a polarization potential of 0.980 volt. In this way the cathodic protection design limits may be established for iron on a good engineering basis at, say, a minimum cathodic protection potential of 0.750 volt and a maximum polarization potential of 1.000 volt. Other design limits would apply to other metals.

Much, however, will depend upon the soil and upon the quality of the coating as shown by the conductance range in the extreme cases illustrated in Discussion Figure 1. There, in the one case of a 3/32-inch glass fabric reinforced coal-tar coating, nearly 600 amperes of current are inadequate on normal standards to protect 130 miles of 14-inch pipe eleven years old, while disrupting the coating by blistering it, destroying the bond of coating to pipe, and by dissolving the glass-fabric reinforcing-mesh to render the coating more porous. In the second case, about 20 amperes of current over-protect to a planned minimum protective potential of 1.000 volt, 335 miles of 6⅝-inch asphalt-mastic coated pipe eighteen years old, with a minimum of abraded coating so far destroyed by the current and with sound coating undamaged at an applied point-of-drainage potential of 4 volts to the copper sulfate electrode in adjacent earth.

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Composition of Corrosion Products Formed on Metals in Steam Condensate★

By R. D. EBERHARDT,(1) E. W. ARNESON(2) and C. E. IMHOFF(3)

Abstract

A study was made of corrosion products formed in steam condensate on the following metals: Carbon steel, cast iron, Admiralty metal, Muntz metal, Aluminum 3S and zinc. The pH of condensate ranged from 5.1 to 9.6 and the test temperatures were 100 F and 212 F. Magnetite was the principal corrosion product formed on carbon steel and cast iron at test conditions. Cuprite was the main corrosion product formed on Admiralty and Muntz metals at both temperatures when the pH was six or less. Metallic zinc corroded to form ZnO over the pH range 5.1-9.6. 3.4.3., 4.6.2

Introduction

THE ORDINARY corrosion products of steel and copper base alloys in commercial condensate consist of various oxides, hydrated oxides and basic carbonates. These are found in steam heated reactor vessels, the lower stages of steam turbines, condenser tubes, heat exchangers, radiators and condensate return lines.

The usual impurities in steam condensates are dissolved gases such as oxygen, carbon dioxide and ammonia.[1] The pH value of the condensate is affected by the ammonia and carbon dioxide concentrations while the oxidation potential is affected by the concentration of dissolved oxygen. These, in addition to temperature, are also factors which determine whether an oxide, carbonate or basic carbonate is the stable end product of a corrosion reaction.

It is not generally appreciated that the condensate collected at the end of a dynamic condensing system, where all the vapors are condensed and all the gases are dissolved, is a composite sample. The actual composition of the condensate varies with the location in the condenser as well as with the operating conditions.

Attempts have been made to determine the cause of corrosion by analyses of the corrosion products.[2, 3] If the correlation of the temperature and composition of the condensate with the products of corrosion were known, the corrosion engineer would be in a better position to determine the conditions and therefore the cause of corrosion.

The purpose of this investigation was to determine which corrosion products are formed on certain alloys in condensate of known composition and temperature. The alloys studied were: carbon steel, cast iron, Admirality metal, Muntz metal, aluminum 3003, and zinc.

Investigation

Procedure

Various metal coupons were placed in contact with 212F steam generated from a small laboratory boiler. The composition of the steam was governed by the composition of the boiler make-up water which was under close chemical control. Heat lost by radiation from the metal coupons caused some of the steam to condense upon the surfaces and corrode the metal. When sufficient corrosion products had formed on the coupons (1-3 months) the deposits were scraped from the surfaces and analyzed by X-ray diffraction.

Metal coupons were also corroded in a humidity cabinet at 100 F and 100 percent humidity. Corrosion products formed under these conditions were similarly analyzed.

Apparatus

A glass steam dome and auxiliaries, as shown in Figure 1, were used to form corrosion products on the metal coupons in 212 F steam. The dome was an inverted two-liter Florence flask which contained a steam outlet to a condenser and eight holes into which rubber stoppers were fitted. Glass hooks on which the metal coupons were suspended were inserted through holes in the stoppers. A glass cold finger was also located in the steam space, and condensate at 212 F was collected in a small funnel and conveyed through glass tubing to the outside of the apparatus for analysis. The dome was located on top of a small laboratory boiler (a brass tube surrounded by an electric heating coil) in which the boiling rate was controlled by a variable transformer connected in series with the coil. Steam, which did not condense on the coupons or cold finger, passed through the dome into a condenser where it was condensed and returned to the boiler make-up reservoir.

A diffuser in the boiler make-up reservoir continuously bubbled a controlled mixture of gases through the make-up water. These gases were dissolved to the limit of their saturation values. The water level in the boiler was adjusted by gravity to the water level in the reservoir.

When several gases such as air and carbon dioxide were used to saturate the boiler make-up, each gas was passed through a rotameter. The ratio of the gases was adjusted to achieve a desired pH. The gases were mixed in a manifold and then passed through the gas diffuser. Ammonia was added to the boiler make-up reservoir as ammonium hydroxide.

The humidity cabinet used to form corrosion products on metal coupons was a 23-cubic foot, insulated, rectangular chamber. An atomizer at one end produced a fog from distilled water and filtered compressed air to maintain the relative humidity at 100 percent. The temperature was controlled at 100 F.

Preparation of Coupons

The dimensions of the metal coupons were one inch by two inches by approximately ⅛ inch. Each coupon had a one-quarter inch hole drilled in one end. Prior to exposure the coupons were

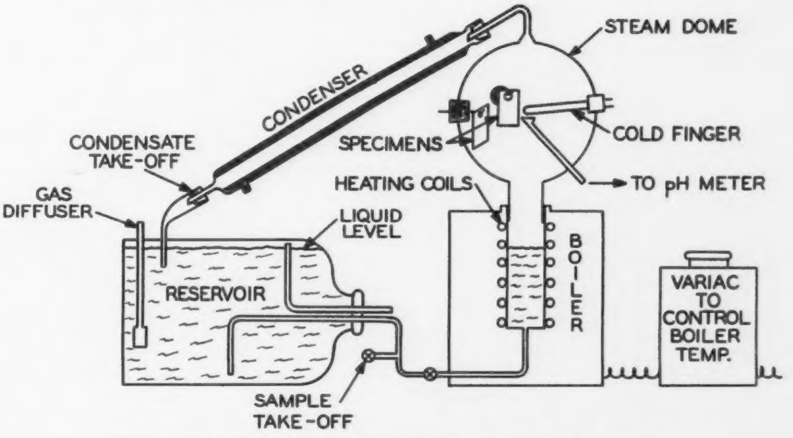

Figure 1—Apparatus used to form corrosion products in 212 F steam condensate.

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TABLE 1—Corrosion Products Formed in Condensate

Gases in Makeup	Air CO_2	Air CO_2	Air CO_2	Air CO_2	N_2	Air NH_3 (2ppm)	Air NH_3 (6ppm)
Temperature of Condensate	100 F	212 F	212 F	212 F	212 F	212 F	212 F
pH of Condensate	6.0	5.1	5.4	6.0	7.2	9.0	9.6
Metals: Carbon Steel	Fe_3O_4 $\gamma Fe_2O_3.$ H_2O	Fe_3O_4	Fe_3O_4	Fe_3O_4	Fe_3O_4	Fe_3O_4 $\alpha Fe_2O_3.$ H_2O	Fe_3O_4
Cast Iron	Fe_3O_4	Fe_3O_4	Fe_3O_4	Fe_3O_4	Fe_3O_4 $\beta Fe_2O_3.$ H_2O	Fe_3O_4 $\alpha Fe_2O_3.$ H_2O	Fe_3O_4 $\gamma Fe_2O_3.$ H_2O
Admiralty Metal	Cu_2O $2\ ZnCO_3.$ $3\ Zn\ (OH)_2$		Cu_2O	Cu_2O ZnO	Zn "E" ZnO		
Muntz Metal	Cu_2O $2\ ZnCO_3.$ $3\ Zn\ (OH)_2$		ZnO Cu_2O	ZnO Cu_2O	Zn "E"	Cu (dezincified)	Cu (dezincified)
Aluminum 3S	αAl_2O_3 $3H_2O$	$\gamma Al_2O_3.$ $3H_2O$	$\gamma Al_2O_3.$ $3H_2O$	$\beta Al_2O_3.$	$\alpha Al_2O_3.$ H_2O		$\gamma Al_2O_3.3H_2O$ $\gamma Al_2O_3.H_2O$
Zinc	$2\ ZnCO_3.$ $3\ Zn\ (OH)_2$		ZnO Zn "E"	ZnO Zn "E"	ZnO	ZnO Zn "E"	ZnO

Note: Blank spaces in the chart indicate that an insufficient quantity of product had formed when the test was terminated.

ground and polished with a number of grit papers (through No. 600), degreased in ether, degreased in acetone, and then rinsed in distilled water. Each coupon was then placed into a controlled atmosphere where it remained until a deposit of sufficient quantity for X-ray diffraction analysis was formed.

Analysis of Condensate

The aqueous film in contact with the metal coupons contained those gases which were taken into solution from the steam at the condensing temperature. To collect samples for determining the quality of the condensate, the cold finger was operated to condense vapor at a very slow rate. Small samples collected continuously from the cold finger were transmitted through a glass tube to a micro-pH cell for direct measurement. The dissolved oxygen concentration of the condensate could not be determined directly because of the limited quantity available for test. Ammonia was determined by direct Nesslerization of the collected sample. It was found that the concentration of ammonia in the condensate was very close to that in the boiler make-up water under the conditions of this experiment.

Analysis of Deposits

Corrosion products formed on the metal coupons were analyzed by X-ray diffraction. The results of these analyses are tabulated in Table 1.

Discussion

Magnetite (Fe_3O_4) was the principal corrosion product formed on carbon steel and cast iron over the range of pH and at the temperatures of this investigation. Lepidocrocite ($\gamma Fe_2O_3 \cdot H_2O$) was a minor product at 100 F. None of the basic ferric oxides were formed at 212 F when the pH was below seven. (These findings somewhat supplement the work of Strauss and Bloom[4] who found lepidocrocite at 25 C and magnetite at 316 C). All three forms of $Fe_2O_3 \cdot H_2O$ (α, β and γ) were formed under different conditions at 212 F when the pH was above seven. The beta form was observed only in oxygen free conditions. Because ferric oxide is not expected to form under oxygen free conditions, this observation should be rechecked in replicate tests.

Cuprite (Cu_2O) was the principal corrosion product formed on Admiralty and Muntz metals at 100 F and 212 F when the pH was six or less. No cuprite was formed at pH values above seven. Muntz metal was dezincified only at the higher pH values (9 and above) caused by the addition of ammonia. Hydrozincite ($2ZnCO_3 \cdot 3Zn(OH)_2$) was formed on Admiralty and Muntz metals at 100 F but not at 212 F.

Hydrozincite was also formed on zinc at 100 F but not at 212 F. Zincite (ZnO) was the principal corrosion product of zinc at 212 F over the pH range investigated. Zinc "E"[5], a corrosion product of unknown composition, was formed on zinc under some experimental conditions at 212 F. The limiting conditions for its existence, however, were not established.

Similarly the conditions for the formation of both zincite and zinc "E" on Admiralty and Muntz metals at 212 F were not determined.

The compositions of the corrosion products of aluminum were too varied to determine any correlation with the factors in this investigation. Because the corrosion products of aluminum are usually amorphous when analyzed by X-ray diffraction[6], other methods of analysis may be more informative.

The corrosion products formed in this investigation were relatively thin films. It is recognized that had corrosion progressed for greater lengths of time, the composition of early corrosion products could have been altered by certain aging reactions.[7]

Conclusions

The following conclusions were reached regarding corrosion products formed in steam condensate at 212 F:

1. Corrosion products most frequently formed on carbon steel and cast iron are magnetite (Fe_3O_4) and three forms of basic ferric oxide (FeOOH, generally written as $Fe_2O_3 \cdot H_2O$). Magnetite is found over the pH range from 5.0 to 9.6. The basic ferric oxides are found more frequently at pH values above 7.

2. Cuprite is the most stable copper corrosion product of Admiralty or Muntz metals at pH values of 6.0 or less. The formation of cuprite is less likely at pH values above 7.

3. The corrosion products of Muntz metal are soluble in two parts per million or more of ammonia and the corrosion takes the form of dezincification.

4. Metallic zinc corrodes to form zinc oxide (ZnO) over the pH range from 5.1 to 9.6. The product zinc "E" may be produced between the pH values 5.1 to 7.2.

It was found in steam condensate at 100 F with a pH value of 6.0 that hydrozincite ($2ZnCO_3 \cdot 3Zn(OH)_2$) is the principal corrosion product of zinc from either metallic zinc, Admiralty metal or Muntz metal.

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DISCUSSION

Comment by L. W. Gleekman, Wyandotte Chemical Corp., Wyandotte, Michigan:

Would not the development of corrosion product films be accelerated by the use of cold fingers made of the metals under study, rather than use of specimens immersed in the vapor with no heat loss other than radiation?

Reply by R. D. Eberhardt and C. E. Imhoff:

A cold finger specimen would undoubtedly be bathed and washed with larger volumes of condensate than the ones used in our experiments. However, this might actually retard the rate of product formation. The constant diluting and washing effect would reduce the metal ion concentration in the water film on the specimen. Under these conditions it would be more difficult to exceed the solubility product of the metal ion compound. The rate of product formation would thereby be reduced.

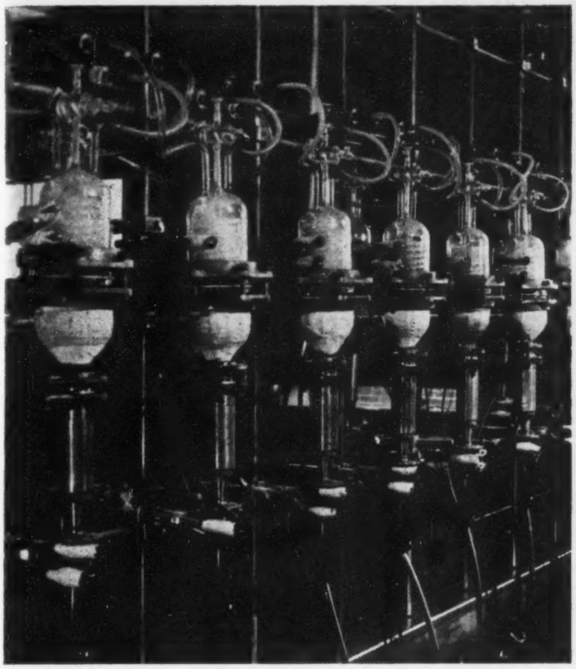

Figure 1—Equipment for evaluating the corrosion resistance of materials under heat flux.

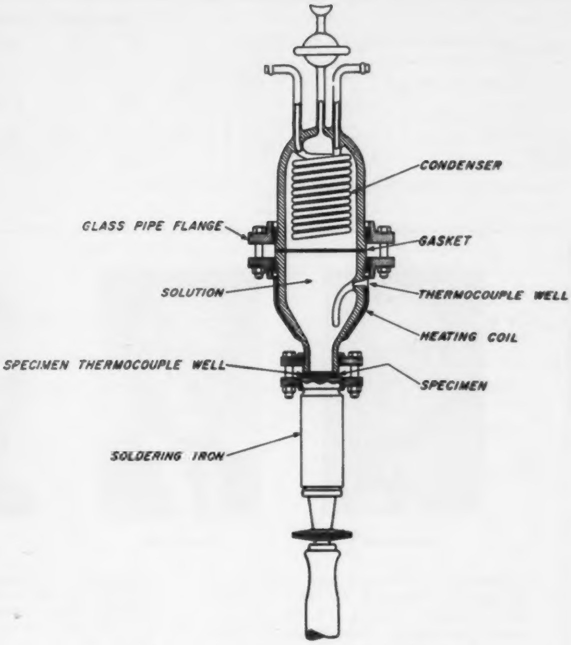

Figure 2—Schematic of equipment pictured in Figure 1.

Corrosion of Metals by Weak Acids Under Heat Transfer Conditions*

By N. D. GROVES, C. M. EISENBROWN and L. R. SCHARFSTEIN

Abstract

Weight-loss measurements were made on six alloys: Types 304L and 316 stainless steels, Carpenter No. 20-Cb, Hastelloy B, Inconel and Monel in solutions of 10 percent, 50 percent and concentrated acetic acid, formic acid and phosphoric acid. A special heat transfer device was used to compare corrosion rates obtained with and without heat transfer. Metal temperatures of 230 F, 257 F and 284 F were studied. Carpenter No. 20-Cb and Hastelloy B consistently showed good corrosion resistance. Of the alloys tested Monel was the most sensitive to the effects of heat throughput.
4.3.2, 6.2.5, 6.3.10, 3.5.9

Introduction

THE SELECTION of a material of construction for a chemical process industry application involves the consideration of chemical, physical and mechanical requirements. A major consideration is corrosion resistance. It is surprising to see how sound principles often are completely ignored by engineers as they scan corrosion rates from published charts and then proceed to the selection of a "corrosion-resistant" material.

Corrosion resistance alone is not sufficient. It is quite illogical to use corrosion data obtained under static conditions when the application involves high velocity, to use data obtained under deaerated conditions when service conditions are aerated, or to use data obtained by simple immersion in the boiling liquid when in the field heat will be passed through the metal to keep the solution boiling. One should use the right table.

In certain cases the best estimate based upon similar data is a practicable solution. Since very little corrosion data are actually available for use when the metal temperature is above that of the boiling solution, the authors decided to investigate the corrosion rates of six common "corrosion-resistant" alloys at different metal temperatures in boiling solutions of three common acids. The alloys selected for study were: AISI Types 304L and 316 stainless steels, Carpenter No. 20-Cb, Hastelloy B, Inconel and Monel. The materials were selected as a minimum number showing important differences in composition which represent alloys finding use in the chemical process industry. The acids are common industrial agents, related to each other in that they are considered relatively weak. Formic acid is a reducing agent; neither acetic or phosphoric acids are regarded as oxidizers or reducers.

Three concentrations of acids were selected: 10 percent, 50 percent and concentrated. Three metal temperatures were 230 F, 257 F and 284 F. Each acid was tested at its atmospheric boiling point except the concentrated phosphoric acid which boils above the temperature limit of the apparatus. The concentrated phosphoric acid was maintained at approximately 210 F for exposures both with and without heat transfer. A comparison was made between simple immersion coupon corrosion rates and those obtained during heat transfer.

★ Submitted for publication August 23, 1960. A paper presented at a meeting of the Northeast Region, National Association of Corrosion Engineers, Huntington, W. Va., Oct. 11-14, 1960.

TABLE 1—Materials Tested

Material	ELEMENT, PERCENT												Heat Treatment
	C	Mn	Si	P	S	Cr	Ni	Mo	Cu	Fe	Co	Cb	
Type 304L	0.02	1.48	0.59	0.012	0.013	18.25	10.45	0.09	.....	Bal.	....	....	Ann. 1950 F, 1 hr., W.Q.
Type 316	0.07	1.70	0.49	0.030	0.023	17.47	13.22	2.15	.....	Bal.	....	....	Ann. 1950 F, 1 hr., W.Q.
C.S. 20-Cb	0.05	0.82	0.48	0.010	0.007	20.60	29.48	2.47	3.46	Bal.	....	0.72	Ann. 1950 F, 1 hr., W.Q.
Hastelloy B	0.01	0.46	0.25	0.014	0.011	0.32	Bal.	26.45	.....	4.83	0.95	....	Ann. 2100 F to 2125 F, 15 min., W.Q.
Inconel	0.04	0.14	0.19	.....	0.007	16.16	76.73	.....	0.05	6.66	....	....	Ann. 1850 F, 10 min., A.C.
Monel	0.13	0.87	0.14	.....	0.007	.....	65.97	.....	31.59	1.27	....	....	Ann. 1700 F, 4 min., A.C.

Scharfstein

Eisenbrown

Groves

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CHARLES M. EISENBROWN is a Corrosion Engineer for The Carpenter Steel Company, Reading, Pennsylvania. He is a member of the National Association of Corrosion Engineers. He has a BS degree in chemistry from Albright College, Reading, Pennsylvania.

NORMAN D. GROVES is Assistant Manager of Research—Chemistry and Physics for The Carpenter Steel Company, Reading, Pennsylvania. Previously he was a Corrosion Engineer for five years at the Hanford Atomic Products operation at Richland, Washington. Mr. Groves is a member of the ASM and NACE. He holds a BS degree in physical chemistry from Huron College, Huron, South Dakota.

TABLE 2—Corrosion Rates (in mpy) of Materials in 10 Percent By Wt Acetic Acid

	Specimen Temperature, Degrees F			
	Without Heat Transfer	With Heat Transfer		
Material	214	230	257	284
Type 304L	<1	<1	<1	<1
Type 316	<1	<1	<1	<1
C.S. 20-Cb	<1	<1	<1	<1
Hastelloy B	3	7	6	4
Inconel	20	28	27	8
Monel	51	580	> 1,000	> 1,000

Experimental

Materials and Specimen Preparation

The corrosion specimens were cut from sheet stock of each of the six materials shown in Table 1. Prior to machining the specimens, the rough-cut samples were annealed as indicated in Table 1.

The finished specimens measured 1½ inches in diameter by 3/16 inch thick. A 3/32 inch diameter hole was drilled into the 3/16 inch edge to the midpoint of the specimens to receive a thermocouple. The final surface preparation was done on a surface grinder to produce uniform parallel sides having approximately a 32 microinch finish. Prior to exposure the specimens were washed with a detergent, rinsed with alcohol, dried and weighed to the nearest 0.0001 gram.

Solutions

The 10 percent and 50 percent concentrations of each acid were prepared by diluting the reagent grade acids with distilled water. The concentrated solution of each acid was the commercially available reagent grade concentration (phosphoric acid—86 percent, acetic acid —99.6 percent, and formic acid—88-90 percent). All acid concentrations were calculated as percent by weight.

Apparatus and Procedure

The equipment used has been previously described[1] and is also shown in Figures 1 and 2. The basic components were manufactured from borosilicate pipe. The lower half of the unit was fabricated from a 3 inch to 1 inch glass pipe reducer, the only modification on which is a thermocouple well. The top half of the unit was manufactured from a 3 inch glass pipe cap which was fitted with a straight bore stopcock and a suitable glass cooling condenser.

The heating source was a standard 550 watt soldering iron, the tip of which was replaced by an aluminum heating block. The current to the soldering iron, which in turn controls the amount of heat to the specimen, was controlled by a Powerstat. Supplementary heat was supplied to the solution contained in the glass pipe reducer by wrapping the reducer with Nichrome wire and asbestos insulation.

The specimen was sealed to the 3 inch to 1 inch glass pipe reducer using fluorocarbon gasket at the specimen-glass interface. The gasket was placed on the 1 inch opening of the vessel followed by the specimen and finally the aluminum heating block which was inserted into a standard Corning glass pipe flange. In order to insure uniform tension on the glass, the bolting operation was done with a torque wrench using a torque of 4 to 5 ft lb. After mounting the specimen to the glass pipe reducer, the glass pipe

TABLE 3—Corrosion Rates (in mpy) of Materials in 50 Percent By Wt Acetic Acid

	Specimen Temperature, Degrees F			
	Without Heat Transfer	With Heat Transfer		
Material	216	230	257	284
Type 304L	130	210	220	250
Type 316	<1	<1	<1	<1
C.S. 20-Cb	<1	2	3	<1
Hastelloy B	5	5	2	2
Inconel	49	44	31	14
Monel	76	120	145	130

TABLE 4—Corrosion Rates (in mpy) of Materials in 99.6 Percent By Wt Acetic Acid

	Specimen Temperature, Degrees F				
	Without Heat Transfer		With Heat Transfer		
Material	230	244	230	257	284
Type 304L		69	260	340	20
Type 316		<1	<1	13	10
C.S. 20-Cb		7	5	2	100 (pits)
Hastelloy B		<1	7	7	3
Inconel		22	36	45	14
Monel	3	1	120	68	220

TABLE 5—Corrosion Rates (in mpy) of Materials in 10 Percent By Wt Formic Acid

Material	Specimen Temperature, Degrees F			
	Without Heat Transfer	With Heat Transfer		
	214	230	257	284
Type 304L	742	> 1,000	570	570
Type 316	10	16	62	73
C.S. 20-Cb	<1	2	8	27
Hastelloy B	10	50	56	80
Inconel	35	73	66	60
Monel	133	640	> 1,000	> 1,000

TABLE 6—Corrosion Rates (in mpy) of Materials in 50 Percent By Wt Formic Acid

Material	Specimen Temperature, Degrees F			
	Without Heat Transfer	With Heat Transfer		
	217	230	257	284
Type 304L	> 1,000	> 1,000	> 1,000	> 1,000
Type 316	39	58	60	84
C.S. 20-Cb	6	9	13	12
Hastelloy B	11	9	5	5
Inconel	61	76	103	115
Monel	87	76	48	100

TABLE 7—Corrosion Rates (in mpy) of Materials in 88-90 Percent By Wt Formic Acid

Material	Specimen Temperature, Degrees F				
	Without Heat Transfer		With Heat Transfer		
	230	217	230	257	284
Type 304L		716	540	500	520
Type 316		10	48	40	48
C.S. 20-Cb		5	4	6	10
Hastelloy B		<1	5	3	7
Inconel		40	50	76	56
Monel	3	1	22	33	50

TABLE 8—Corrosion Rates (in mpy) of Materials in 10 Percent By Wt Phosphoric Acid

Material	Specimen Temperature, Degrees F			
	Without Heat Transfer	With Heat Transfer		
	214	230	257	284
Type 304L	<1	<1	2	4
Type 316	<1	10	13	9
C.S. 20-Cb	<1	2	3	4
Hastelloy B	22	8	4	7
Inconel	<1	36	80	100
Monel	10	42	40	115

TABLE 9—Corrosion Rates (in mpy) of Materials in 50 Percent By Wt Phosphoric Acid

Material	Specimen Temperature, Degrees F			
	Without Heat Transfer	With Heat Transfer		
	230	230	257	284
Type 304L	8	13	3	> 1,000
Type 316	6	39	4	22
C.S. 20-Cb	17	20	4	59
Hastelloy B	3	5	10	14
Inconel	176	290	900	> 1,000
Monel	4	120	310	870

TABLE 10—Corrosion Rates (in mpy) of Materials in 86 Percent By Wt Phosphoric Acid

Material	Specimen Temperature, Degrees F				
	Without Heat Transfer		With Heat Transfer		
	230	208.4	230	257	284
Type 304L		> 1,000	22	> 1,000	> 1,000
Type 316	9	16	110	215	35
C.S. 20-Cb		2	2	19	24
Hastelloy B		<1	1	2	2
Inconel	4	46	400	> 1,000	> 1,000
Monel		1	83	840	> 1,000

cap was bolted to the reducer using a standard 3 inch fluorocarbon gasket and two standard 3 inch glass pipe flanges. For this assembly a torque of 2 to 3 ft lb was used.

One hundred and fifty milliliters of corrodent were added through the straight bore stopcock at the top of the unit by using a graduated cylindrical separatory funnel fitted with a length of small diameter polyvinylidene chloride tubing. The corrodent was renewed every 48 hours by draining through the stopcock using the plastic tubing and a water aspirator.

While bringing the metal specimen to the desired temperature, the stopcock was opened slightly until operating conditions were reached. By varying the current to the soldering iron, the temperature of the metal specimen was raised to the desired temperature. Supplementary additions of heat were supplied to the corrodent by varying the current to the Nichrome heating coil. The temperature of the metal specimen and the corrodent were continuously recorded by means of thermocouples and a strip chart recorder. Metal specimen temperatures were maintained at plus or minus 4 F of the desired operating conditions.

Following the 48-hour and 96-hour test durations, the metal specimens were removed from the apparatus and weighed. From the difference between the initial and final weight of the specimen and the area of exposed surface, a corrosion rate was calculated using the standard formula:

$$\frac{3{,}451{,}000 \text{ x wt loss (g)}}{\text{Exposed Area (cm}^2\text{) x Time (hr) x Density (g/cm}^3\text{)}}$$

= mpy (mils penetration per year)

Discussion

The corrosion rates obtained during the course of this work are summarized in Tables 2 to 10. The best materials, under heat transfer conditions, were Carpenter No. 20-Cb and Hastelloy B. Type 316 stainless steel can also be recommended in certain cases.

A comparison can be made between the rates at approximately 220 F with and without heat transfer. Monel appears to be the most sensitive material, of those tested, to the effect of heat transfer. Carpenter No. 20-Cb did not show noticeably different rates as a result of heat transfer. Neither did Hastelloy B. The major effect of heat transfer at a given temperature may be associated with the physical nature of the oxide films, alloys with high chromium and/or molybdenum contents surviving the best. This may only be an effect of the acids studied, but additional work along these lines is contemplated.

Another observation deserving comment is that one acid has a much greater effect on increasing corrosion than another as the metal temperature is raised in 27 F increments. Formic acid is particularly effective; phosphoric acid is similar. Thus, if one does not have corrosion data for a particular alloy undergoing heat transfer in formic or phosphoric acid, a serious error may be made by estimation based on immersion test data.

After performing hundreds of tests with the apparatus described here, the authors have concluded that the reproducibility of the corrosion rate calculations is probably better than plus or minus 50 to 100 percent throughout the range of 1 to 1000 mils per year. Although this is a large deviation, it is sufficiently small to rank materials since the engineer is generally more interested in knowing whether the rate is 2 or 20 mils per year and not 2.0 or 2.2 mils per year. One possible source of error is localized attack at or beneath the fluorocarbon gasket. This type of attack is seen occasionally; in such cases results obtained when it is evident are discarded.

The "crevice" or "concentration cell" attack is caused either by loose or uneven gasketing or is associated with the environment. Corrosion at the gasket-metal interface did not occur in performing this work using acetic, formic and phosphoric acids. Sulfuric acid is an environment that frequently causes localized attack at the gasket with stainless steels.

Conclusions

On the basis of six alloys tested in three concentrations of acetic, formic and phosphoric acid where the metal temperature was maintained above that of the solution, it is concluded that:

1. Carpenter No. 20-Cb and Hastelloy B most consistently showed the best corrosion resistance regardless of metal temperature.

2. Monel was the most variable alloy regarding corrosion behavior as a function of metal temperature.

3. Formic acid has a great tendency to increase corrosion rates as the metal temperature is raised. Acetic acid has the least tendency.

4. Incorrect corrosion rates would be obtained if one used simple immersion data to extrapolate to higher metal temperature.

Acknowledgments

The authors wish to thank The Carpenter Steel Company for permission to publish this work and to acknowledge with gratitude the extra effort expended by the Technicians of the Corrosion Research Laboratory to maintain and operate the equipment over a continuous four-month period.

Reference

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Any discussion of this article not published above will appear in June, 1961 issue.

Aluminum, Aluminized Steel And Galvanized Steel In Severe Coastal Environments*

By THOMAS A. LOWE

About the Author

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Abstract

Aluminum, aluminized steel and galvanized steel pole line hardware were exposed in a severe marine environment near Kahuku Point, Oahu, Hawaii for three years. Variables included aluminizing process and type of base steel. Evaluation of samples after three years' exposure indicates: (1) the superior performance of all-aluminum hardware, (2) the improved corrosion resistance of aluminized steel to that of galvanized steel, and (3) the relationship between the thickness of aluminum coatings and the service life of aluminized steel hardware. Thicker aluminum coatings result in longer life. 2.2.2, 4.2.5, 6.4.2, 5.3.4

Introduction

RISING COSTS of installing, maintaining and replacing outdoor equipment have heightened the importance of design and material selection in reducing corrosion damage. Aluminum and its alloys have received much attention by designers because of their good corrosion resistance, their long life and good maintenance record. At the same time, advances in the technology of aluminizing have led to improved coatings at competitive costs. These advances have raised the question of the comparative corrosion resistance of aluminized steel and aluminum.

In order to evaluate a commercial material which would lend itself to valid comparison, pole line hardware was selected. Such hardware is available in several alloys and with a variety of coatings. Accordingly, an evaluation of the corrosion performance of these hardware variables was initiated to:

1. Compare the relative corrosion resistance of steel which had been aluminized by two different hot-dip processes.
2. Compare the corrosion performance of aluminum, aluminized steel and galvanized steel in marine environments.
3. Determine the influence of base material on the performance of aluminum and zinc coatings.

Of supplemental interest were the effects of treated wood on the materials and the relative susceptibility of aluminized and galvanized threaded items to galling and seizing.

Test Location and Installation

The site chosen for this study was in Hawaii, on the windward shore of Oahu. Severe marine atmospheres exist in this area because strong trade winds blow from the ocean where waves break constantly over shallow, coral-covered reefs.

The materials were installed on February 15, 1956. The hardware items on the various crossarms were staggered slightly to minimize drippage of moisture or corrosion products onto the pieces mounted on lower crossarms. Four crossarms, with identical sets of hardware, were included to allow the exposure periods of one, three, five and ten years. The completed installation is shown in Figure 1.

Material Variables

Though experience has proven most aluminum alloys to be acceptable materials of construction in marine atmospheres, a direct comparison of all-aluminum hardware with other materials was desired. Commercially available aluminum items in both cast and wrought alloys were included. Analyses showed the aluminum hardware to consist of the following alloys:

Cast secondary rack	Pin - 6061, Al-Mg-Si alloy Body - 356, Al-Si-Mg alloy
Forged secondary rack	6061, Al-Mg-Si alloy
Clevis	Channel - 6063, Al-Mg-Si alloy Pin - 6061, Al-Mg-Si alloy
Clamp-on insulator pin	355, Al-Si-Mg alloy
Pole step	6061, Al-Mg-Si alloy
Pole band	6061, Al-Mg-Si alloy
Crossarm brace	5086, Al-Mg-Mn alloy

Table 1 gives chemical composition limits.

Hardware coated by two hot-dip processes were included in the study. The two processes, A and B, differed in several ways. In Process A,

1. Alloy K726 with 2.5 percent nominal silicon was used as a coating alloy.
2. An aqueous halide served as the fluxing solution.
3. Coating was applied in a laboratory operation.

This process was used on both plain carbon and low-alloy steel. The resultant coatings had a uniform thickness of aluminum over a somewhat thinner, but uniform iron-aluminum interfacial layer⁽¹⁾. Typical coating structure is shown in Figure 2. In Process B,

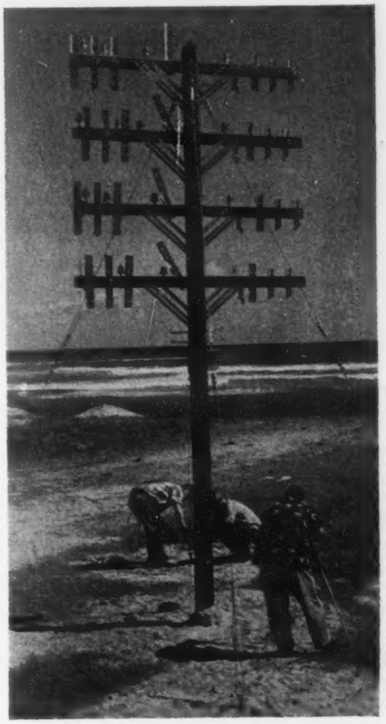


Figure 1—The completed test installation.

1. An 1100-type alloy, commercially pure aluminum with no silicon addition, was used.
2. Fluxing was done in a molten salt bath.
3. Coating was applied in a commercial scale operation.

Only plain carbon steel was coated by this process. Though over-all coating

TABLE 1—Composition of Aluminum Hardware Alloys[1]

Alloy Type	Element						
	Silicon	Iron	Copper	Manganese	Magnesium	Chromium	Zinc
355......	4.5 —5.5	0.40	1.0 —1.5	0.20	0.40—0.60	....	0.10
356......	6.5 —7.5	0.35	0.20	0.10	0.25—0.40	....	0.10
5086......	0.40	0.50	0.10	0.20—0.70	3.5 —4.5	0.05—0.25	0.25
6061......	0.40—0.80	0.70	0.15—0.40	0.15	0.8 —1.2	0.15—0.35	0.25
6063......	0.20—0.60	0.35	0.10	0.10	0.45—0.90	0.10	0.10

[1] Elements shown in percent; values are maximum unless shown as a range.

⁽¹⁾ Covered by patent rights of Kaiser Aluminum & Chemical Corporation.

★ Submitted for publication August 22, 1960.

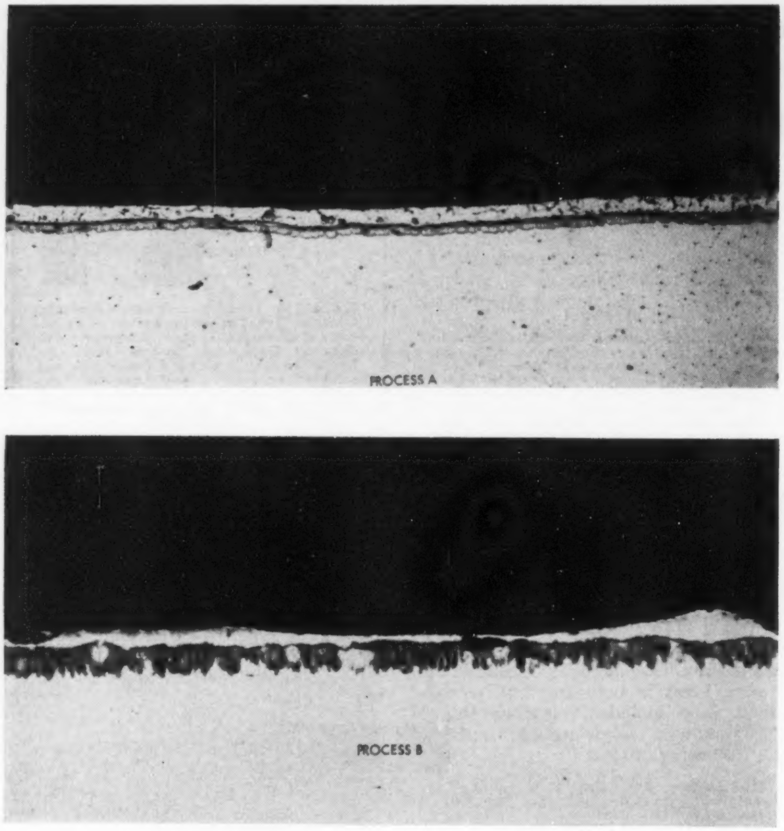


Figure 2—Process A aluminized carbon steel before exposure; Process B aluminized carbon steel before exposure. 50X reduced to 24X, ½ percent HF etch.

thicknesses produced by the two processes were equivalent, there were important differences in structure. Process B coatings consisted of a much thicker intermetallic layer and thinner aluminum layer than exhibited by those of Process A. Typical coating structure is shown in Figure 2.

Galvanized carbon and low-alloy steel hardware included readily available, standard pole line hardware material. These pieces were all commercially galvanized by the hot-dip method.

Evaluation Procedure

At the end of each scheduled exposure period, a complete cross arm assembly was removed and returned to the laboratory. Every effort was made to insure careful handling of the equipped crossarms. In the laboratory, a visual check of the hardware was made to confirm observations made at the site. A complete photographic record of hardware appearance was maintained. All readily accessible nuts were loosened with a torque wrench to evaluate corrosion damage as well as galling and seizing characteristics.

Random samples from each of the major items were selected for metallographic examination. The presence of coating layers was checked. Both coating and interfacial layer thicknesses were measured metallographically. Total coating thickness was checked by means of an Aminco-Brenner Magne Gage. Any damage to the underlying base metal was appraised. Photomicrographs were made of typical sections as well as of unusual areas or areas which revealed some interesting coating characteristic.

An attempt was made to evaluate not only the performance of the material but also the practical service life of the item to the operating utility.

Test Results

The data presented (Table 2) summarize the evaluation of material exposed for one and three years. The results of one year's exposure represented a trend in corrosion performance which continued. For that reason, only general comments will be made relative to them. Most descriptions concern three-year results. Appearance of some of the three-year material is shown in Figures 3, 4, and 5.

Aluminum Hardware

The performance of all-aluminum hardware after three years' exposure has been excellent. In fact, all-aluminum hardware has proven to be the most corrosion resistant of the materials exposed in these tests. Random pitting was noted on both lee and windward surfaces with attack being somewhat more severe on the lee side. This difference is not uncommon for aluminum where natural washing of windward surfaces by rain or wind-borne spray is more frequent. The depth of pitting was insignificant, maximum being 5 mils. Corrosion products from steel items to the windward side

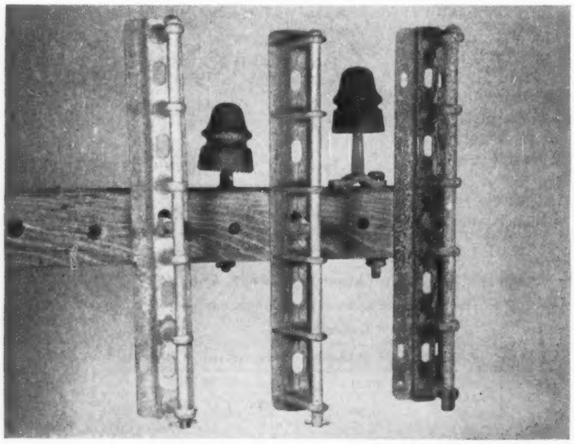


Figure 3—Secondary racks after 3 years' exposure. Left to right: Process A aluminized low-alloy steel, cast aluminum, galvanized low-alloy steel.

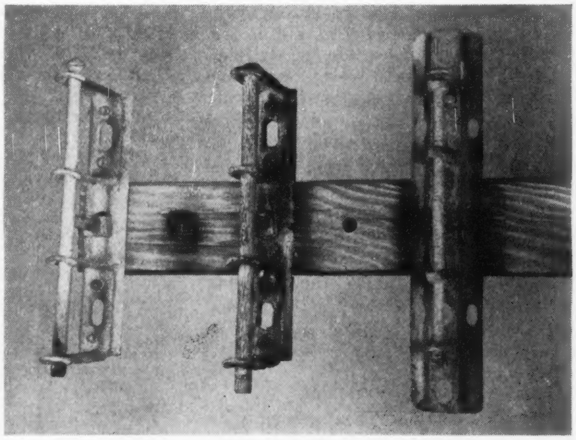


Figure 4—Carbon steel secondary racks after 3 years' exposure. Left to right, Process A aluminized, galvanized, Process B aluminized.

were deposited on the surface of adjacent aluminum pieces influencing their color and appearance but having no effect on performance.

Corrosion was accelerated at those surfaces mating with galvanized steel fasteners after the zinc coating had been expended. This is to be expected because of the electro-chemical relationship of aluminum and zinc, and of aluminum and steel. The more electro-negative character of zinc results in protection of aluminum, as well as steel, initially. However, after the zinc is consumed, the aluminum is preferentially attacked to galvanically protect the steel. The use of aluminized threaded ware resulted in far superior performance of the aluminum faying surface.

Aluminized Steel—Process A

There appears, at this time, to be no advantage in the performance of aluminum-coated, low-alloy steel over similarly coated carbon steel. The evaluation of these variables will therefore be grouped together in discussion.

The aluminum coatings were generally unaffected, and surfaces, with few exceptions, were bright and clean. Threaded pieces aluminized by Process A performed much better than the other threaded items. They were not as consistently bright as the pieces with flat surfaces; here, small dark gray splotches were scattered over the surface. Few points of rust were noted on the threads. The exceptions in coating performance were the result of improper installation. For example, brass cotter pins were used through error with the secondary rack rods, and a galvanized steel bolt was used in one case as a substitute for an aluminized threaded assembly whose threads were stripped.

A metallographic study revealed that Process A coatings had not been significantly affected by the three years' exposure. Over the majority of the surfaces, the aluminum layer was intact and the iron-aluminum intermetallic layer had not been penetrated. This can be seen in Figures 2 and 6.

Coating thickness measurements indicated that no appreciable change had taken place in overall thickness as a result of three years' weathering. Such a conclusion is evident from Table 2 as well as from the photomicrographs (Figures 2 and 6) of typical sections.

Aluminized Steel—Process B

Items aluminized by Process B lost much of the aluminum layer after one year's exposure. Points of light rust were evident on a dark gray surface. The dark appearance was attributed to the color of the weathered iron-aluminum intermetallic layer. After three years, there was no evidence of aluminum alloy remaining. The intermetallic layer, however, displayed good resistance. Approximately 25 percent of the surface was spotted with rust stains. However, insulator rods were free in the rack, and the serviceability of the hardware was not affected.

The photomicrographs of the Process B aluminized carbon steel section in Figure 2 show how thin the aluminum layer was before exposure. A typical section from Process B hardware after three years' exposure, Figure 6, indicates that no aluminum layer remains. The intermetallic layer, of inconsistent thickness, was generally intact with only random damage evident.

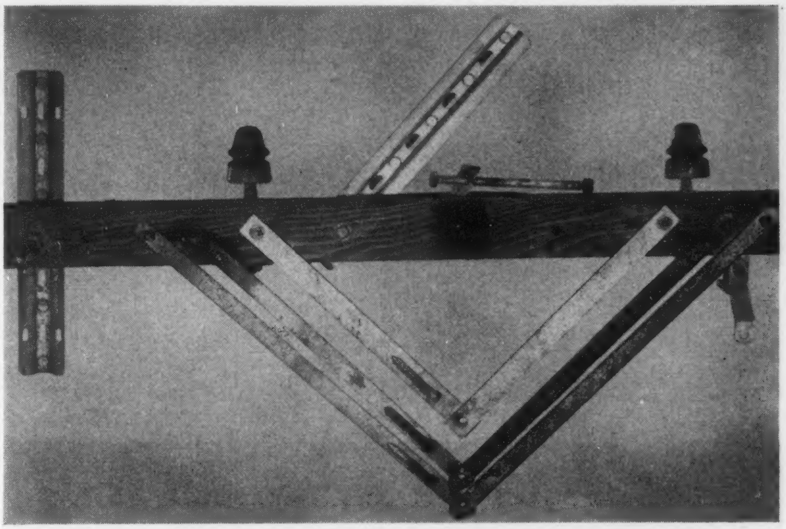


Figure 5—Crossarm braces after 3 years' exposure. (All-aluminum rack extends above.)

Left (Top to Bottom)	Right (Top to Bottom)
All-aluminum	All-aluminum
Process A carbon steel	Galvanized carbon steel
Process A low-alloy steel	Galvanized low-alloy steel

TABLE 2—Average Thickness of Representative Coatings—Mils

	Unexposed[1]		After 3 Years' Exposure[2]
	Average	Range	
Secondary Racks:			
Galvanized carbon steel	2.66	1.9—3.9	*
Galvanized low-alloy steel	6.36	4.7—8.9	*
Process A aluminized carbon steel	3.19	2.3—4.8	3.33
Process A aluminized low-alloy steel	3.42	1.8—6.4	2.54
Process B aluminized carbon steel	2.34	2.0—2.7	2.34
Crossarm Braces:			
Galvanized carbon steel	3.53	2.5—4.8	*
Galvanized low-alloy steel	5.40	3.7—7.3	*
Process A aluminized carbon steel	2.70	1.7—4.2	2.36
Process A aluminized low-alloy steel	2.67	2.3—3.3	2.66

[1] Average determined from measurement of 3-4 pieces of hardware.

[2] Average thickness determined from measurements on one piece of hardware. There is no recorded average coating thickness for these specific pieces before their exposure. Some anomalies in their individual average thicknesses and those of unexposed groups can be expected.

* No zinc remaining.

Galvanized Steel

Though first year results indicated a superiority of zinc coatings on low-alloy steel over those on carbon steel, two additional years' exposure nullified any significant difference. All galvanized hardware failed. The galvanized coatings, in nearly all cases originally thicker than the aluminum coatings, had disappeared within three years. Typical original coating thickness is shown in Figure 7. Threaded ware were badly frozen due to corrosion. Secondary rack rods could not be moved. The steel base material was beginning to pit, as shown in the photomicrograph of a typical section from a galvanized low-alloy steel crossarm brace (Figure 8).

Other Observations

Aluminum-to-aluminized steel mating surfaces were performing satisfactorily. At those points where aluminum and galvanized steel were in contact, corrosion of the aluminum member was accelerated after the zinc coating had been expended.

No appreciable increase in attack resulted with any of the hardware in contact with the creosoted pole or pentachlorophenol treated crossarm. This substantiated previous laboratory tests and experience with aluminum roofing and fencing.

Galling and seizing was not a problem with Process A aluminized steel. The tightly adherent corrosion product on Process B surfaces made threaded hardware of that material difficult to break free. Torque values for the galvanized steel fittings fell between those for the two aluminizing processes. Their consistent nature indicated a uniformity of corrosion and of the bond strength between steel and its corrosion product.

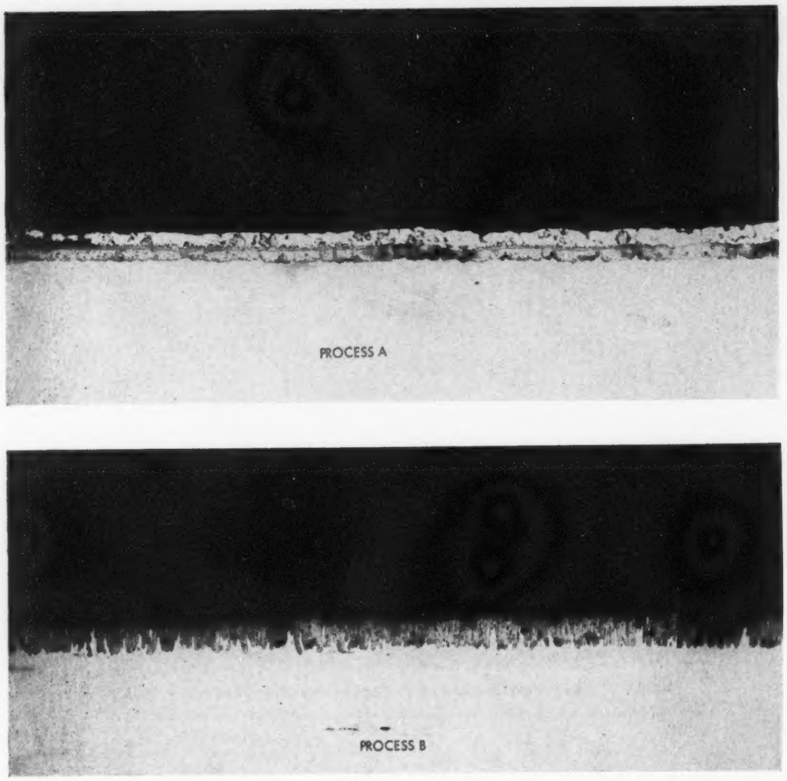


Figure 6—Process A aluminized carbon steel after 3 years' exposure; Process B aluminized carbon steel after 3 years' exposure. 50X reduced to 24X, ½ percent HF etch.

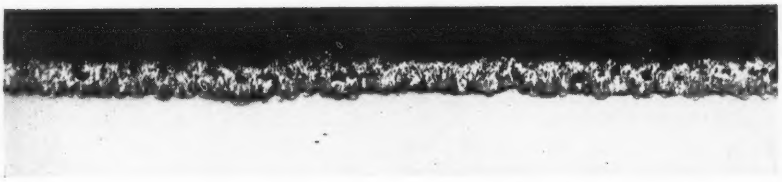


Figure 7—Galvanized low-alloy steel brace before exposure. Etch: ½ percent HF, 50X reduced to 24X.

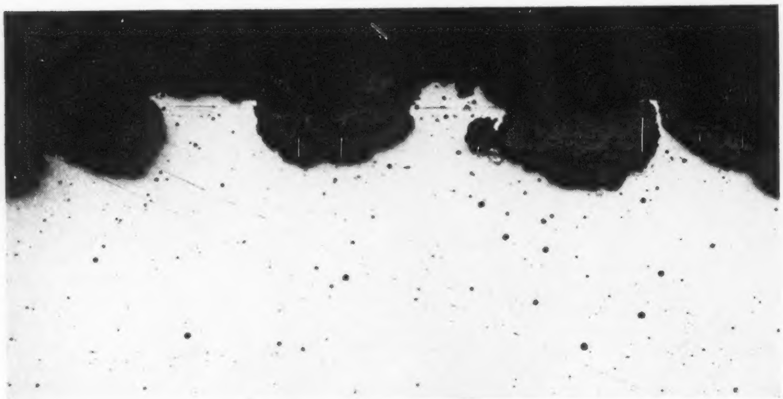


Figure 8—Same coating and base material as Figure 7 after 3 years' exposure. Etch: ½ percent HF, 50X reduced to 24X.

Discussion

It is evident that aluminum hardware is well suited for marine environments. To take advantage of this performance, however, it is necessary that such hardware be installed with aluminum or aluminized steel fasteners.

Hardware aluminized by Process A is superior in corrosion resistance to that aluminized by Process B. A comparison of the coatings produced by the two processes explains the reasons for this difference in performance. The overall coating thickness produced by each process was comparable, but Process A provided a thicker, more uniform aluminum layer.

The corrosion resistance of aluminized steel is primarily dependent upon the thickness and uniformity of the aluminum layer. Discontinuities in this layer expose a less resistant substrate to the atmosphere. Subsequent rusting at these pinholes results in the surface discoloration which so often has been interpreted as complete coating failure. In hot-dipped coatings, the occurrence of pinhole "holidays" decreases with increased coating thickness.

Though processing variables affect the aluminum layer thickness, it is also influenced to some extent by the coating alloy used. The use of coating alloys, which include inhibitors to limit solution of steel in the coating, provide more uniform and thinner intermetallic layers as well as thicker aluminum layers.

Structure of this intermetallic layer also may have some influence on corrosion performance. For example, a study of the intermetallic layer produced by Process B indicates "fingers" of steel which occasionally penetrate to the surface. These are the irregularly shaped areas of the base metal which extend into the iron-aluminum layer. It is believed that the corrosion of the "fingers" contributed to the rust discoloration noted on the surfaces of Process B hardware. If attack on these exposed "fingers" continues, penetration of the intermetallic layer will result, along with subsequent corrosion of the base material. In the same way, corrosion of the "fingers" of intermetallic, which extend to the coating surface can cause premature rust discoloration.

Sufficient thickness of the aluminum layer, regardless of the coating alloy or process used, would insure the continuity to prevent premature rusting or discoloration of aluminized hardware. The corrosion resistance of hardware aluminized by the two processes included in this study would probably have been comparable if this thickness had been equal, assuming that no residual halide contamination from the molten flux bath remained on Process B material.

Galvanized steel, regardless of base material, followed the performance of similar hardware which has been used in the general test vicinity. It was not satisfactory in severe marine atmospheres and was out-performed by all-aluminum and aluminized steel hardware.

Corrosion Product Films Formed on Aluminum In High Temperature Water*

By D. F. MacLENNAN

Abstract

A study was made of the behavior of aluminum alloys exposed to 300 C high purity water for short periods of time. The corrosion products formed were examined by means of optical and electron microscopy. Results show that corrosion resistance is associated with the distribution of second phase particles in the alloys. The alloy, which had the best corrosion resistance, contained the most uniform distribution of cathodic second phase particles; the corrosion product film of this alloy contained a corresponding distribution of irregularities. It is suggested that the second phase particles modify the film in such a way as to increase its protective qualities. 6.4.2, 4.6.5, 3.2.3

Introduction

INTEREST IN the corrosion of aluminum in 300 C water arises in connection with the use of aluminum in water-cooled nuclear power reactors. Because of its low cost and low neutron capture cross section, aluminum is a desirable material for use in power reactors. However, the common aluminum alloys corrode rapidly in water at temperatures above 200 C. Ordinary 2S aluminum (99.0 percent +) behaves well up to about 200 C but above this temperature corrosion occurs very rapidly. At these temperatures pure aluminum (99.99 percent) corrodes more rapidly than 2S.

Draley and Ruther[1] found that small additions of elements, cathodic to aluminum, such as iron and nickel, changed the localized rapid attack to a uniform corrosion of the surface. Krenz[2] has produced two Al-Ni-Fe-Si alloys which behave well in 300 C water. The compositions of these are given in Table 1. After a period of comparatively rapid attack, the corrosion of these alloys slows down and proceeds at a linear rate. The 157 alloy behaves slightly better than the 155.

Many studies have been undertaken to determine the mechanism by which the alloying elements improve the corrosion behavior. A better understanding of this mechanism would likely lead to further improvements in the alloy composition.

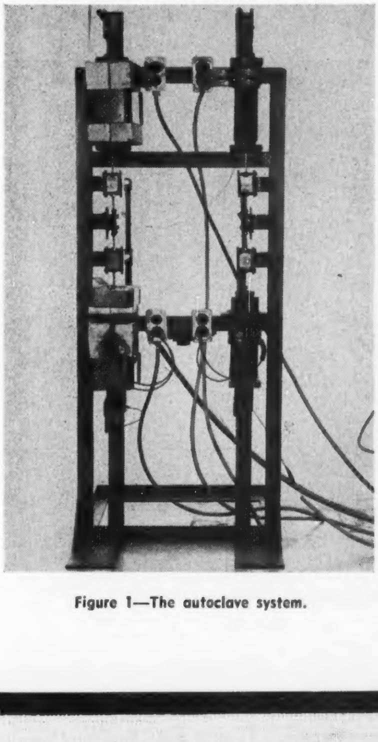
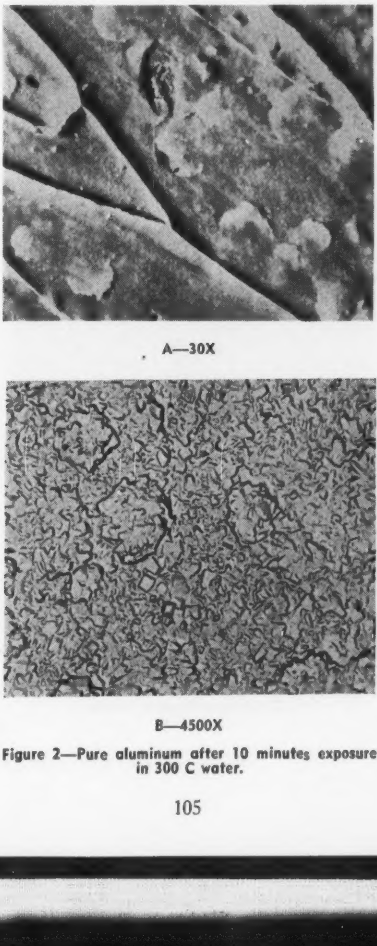


Figure 1—The autoclave system.

About the Author

D. F. MacLENNAN is a member of the Physical Chemistry Section, Naval Research Establishment, Dartmouth, Nova Scotia. He is engaged in a study of the properties of oxide films formed on aluminum and aluminum alloys. He received BSc (1949) and MSc (1951) degrees from Dalhousie University and a PhD (1954) degree from McGill University.

Draley[1] has proposed that, at high temperatures, hydrogen atoms diffuse into the metal, combine at imperfections in the lattice, and with increasing pressure rupture the metal exposing a fresh surface to the water. The second phase particles in the nickel-bearing alloys act as local corrosion cathodes where hydrogen atoms combine to form molecules and are thus prevented from diffusing into the metal.



A—30X

B—4500X

Figure 2—Pure aluminum after 10 minutes exposure in 300 C water.

Tragert's experiments[3] with high purity aluminum led him to suggest an attack brought on by the deformation of the metal due to intrusion of corrosion product at grain boundaries. From work with aluminum-1 percent nickel alloy, Tragert[4] concludes that rather than function as local cathodes, the second phase particles alter the nature of the corrosion film making it less permeable to the diffusing reactants.

Troutner[5] studied the structure of oxide films formed on aluminum alloys after reasonably long exposures to high temperature water. He did not observe any relationship between the original metal surface, the inner corrosion product surface, and the outer corrosion product surface. In the present work, samples of pure aluminum, 2S, 155 and 157 alloys were corroded in water at 300 C for short periods of time in stainless steel autoclaves. After this exposure, the corrosion product was examined by means of optical and electron microscopy. The work differs from Troutner's in that attention is focussed on the initial stages of reaction, and hence the experimental details differ slightly.

The results of this investigation indicate that a definite relationship exists between these surfaces.

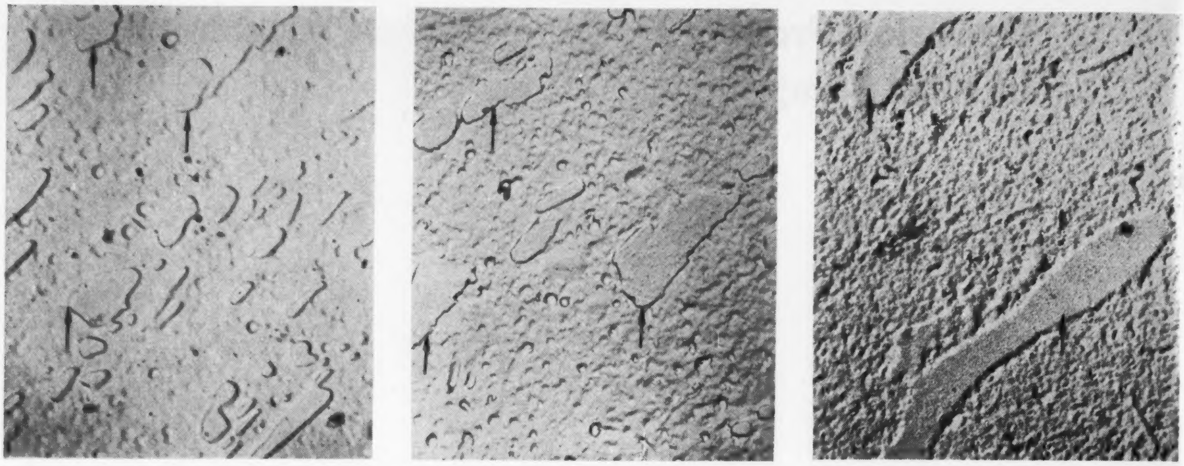
Experimental

The samples were exposed to high temperature pure water in the autoclave systems shown in Figure 1. This figure shows two similar systems mounted on a stand. The one on the left has the moulded insulation in place, while the other has the insulation removed to show

TABLE 1—Aluminum Alloy Compositions

Alloy	Element, Percent		
	Ni	Fe	Si
155..........	0.5	0.5	0.2
157..........	2.0	0.5	0.2

★ Submitted for publication April 12, 1960.

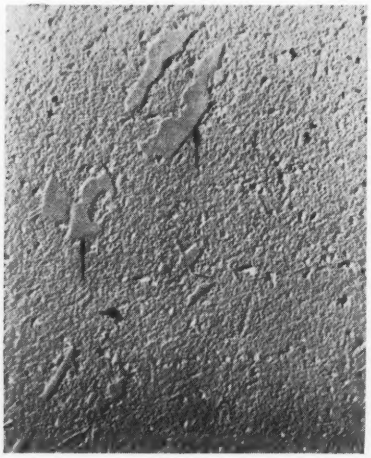


A—157 alloy, 4500X

B—155 alloy, 4500X

C—2S alloy, 4500X

Figure 3—Uncorroded alloys showing second phase particles as indicated by arrows. Metal etched in 1 percent NaOH for five seconds at room temperature before replicating.

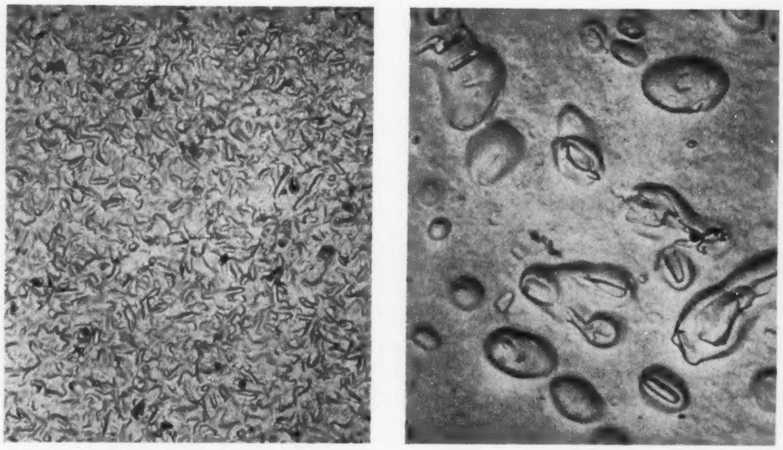


D—2S alloy, 1500X

the stainless steel reaction vessels. Each vessel had a volume of approximately 110 mls and was fitted with a thermocouple for recording the temperature.

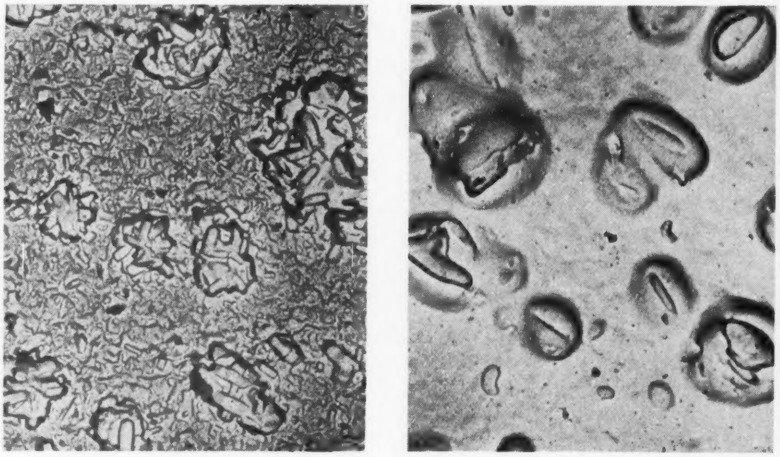
The samples were mechanically polished cylinders $\frac{3}{8}$-inch in diameter and $\frac{5}{16}$-inch long. These were mounted on a stainless steel rack and placed in the lower autoclave. This vessel was then filled with argon, sealed by the valves at either end, and heated so as to anneal the samples for two hours at 344 C. Water was placed in the upper autoclave and heated to the test temperature. After annealing, the lower vessel was brought to the test temperature and the water was transferred by opening the center valve. This procedure put high temperature water on the samples at zero time. At the end of the test time the water was released, as steam, by opening the bottom valve.

Since the actual corrosion films on the samples were not suitable for examination in the electron microscope, replicas of the film surfaces were prepared. The outer surface of the oxide was coated with plastic, which was stripped from the oxide when dry and used as a mould



A—Outer surface, 1 minute

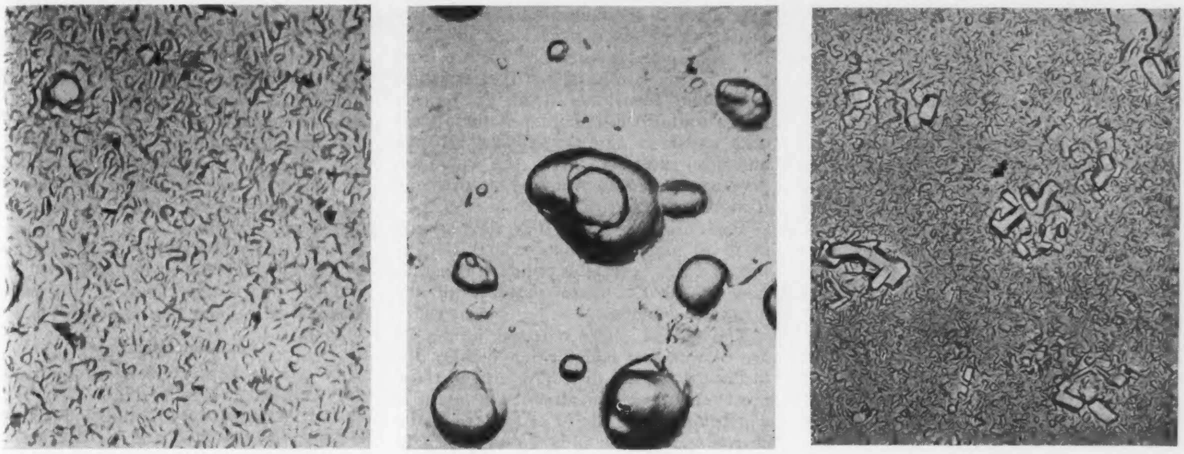
B—Inner surface, 1 minute



C—Outer surface, 5 minute

D—Inner surface, 5 minute

Figure 4—157 alloy after exposure in 300 C water. All photographs at 4500X.

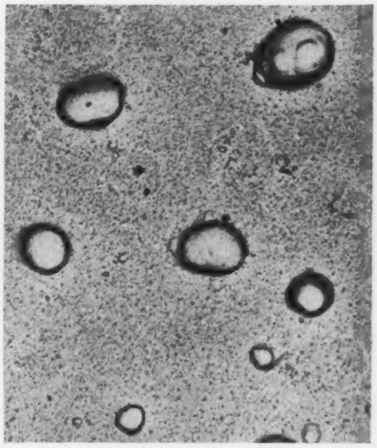


A—Outer surface, 1 minute

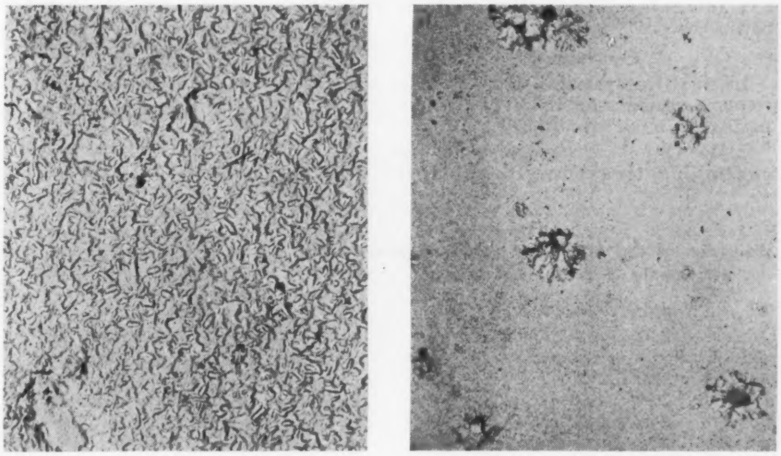
B—Inner surface, 1 minute

C—Outer surface, 5 minute

Figure 5—155 alloy after exposure in 300 C water. All photographs at 4500X.

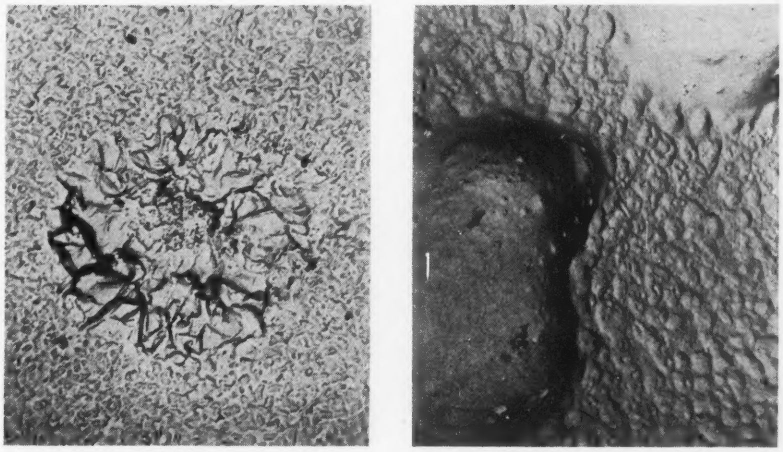


D—Inner surface, 5 minute



A—Outer surface, 1 minute, 4500X

B—Outer surface, 5 minute, 1500X



C—Outer surface, 5 minute, 4500X

D—Inner surface, 5 minute, 4500X

Figure 6—2S aluminum after exposure in 300 C water.

onto which a thin film of carbon was evaporated. The carbon replica was isolated, by dissolving the plastic and then shadowed with gold to give a three dimensional effect. In order to examine the inner surface of the oxide the sample was mounted in a plastic block and most of the metal was cut away. The remaining metal was then dissolved in either dilute sodium hydroxide or iodine-methanol solution, which does not attack the oxide. This procedure exposed the inner face of the corrosion film which was then replicated in a manner similar to that used on the outer surface.

Test Results

The inner and outer surfaces of the corrosion films on many samples have been examined and typical examples are shown here. Figure 2 shows the outer surface of the corrosion film on pure aluminum after ten minutes exposure in 300 C water. The low magnification picture, A, shows how the film has cracked along lines of stress. The high magnification picture, B, shows relatively uniform crystals with patches which appear to have bulged above the surface. Figure

3 shows the size and distribution of second phase particles in the uncorroded alloys. Figures 4-6 show the oxide films formed on the alloys in 300 C water, inner and outer surfaces, after various exposures.

In the nickel-bearing alloys the second phase particles and film irregularities are smaller and more uniformly distributed than in the 2S, with the 157 alloy having a more uniform distribution than the 155. In the 2S samples the second phase particles are large and relatively far apart. The irregularities in the oxide films are also large and far apart.

Discussion

The pictures of the corrosion product on the pure aluminum suggest that corrosion is proceeding according to Tragert's[3] film cracking hypothesis.

After one minute exposure the outer surface of the oxide film on the 157 alloy is crystalline and appears to be quite uniform. The inner surface is amorphous with mounds on the film which correspond to depressions in the metal. After five minutes exposure the outer surface contains clusters of larger crystals and their size and distribution correspond to the mounds on the inside of the film. Many of the mounds contain particles which are similar in shape to the second phase particles in the metal and it is believed that these actually are the particles from the metal. This suggests a model in which the second phase particle and the aluminum in the immediate area form a local cell in which the aluminum is corroded rapidly and the reaction continues until the particle is isolated from the metal. It appears that a uniform distribution of these local cells causes a more uniform corrosion and also possibly alters the corrosion product film in such a way as to make it more protective.

The corrosion of the 155 alloy results in fewer mounds and large crystal clusters and this correlates with the distribution of second phase particles in the uncorroded metal.

The second phase particles in the 2S are of different composition besides differing in size and distribution from those in the 157 and 155 alloys. The mounds which appear on the inner surface of the corrosion product film do not contain second phase particles. Thus it is suspected that a different mechanism may be acting in this case. However, if the mechanisms are similar, the modification of the corrosion product film due to the second phase particles would be much less effective because of the large size and irregular distribution of these particles.

Conclusions

1. On all samples the entire surface becomes coated with oxide almost immediately.

2. (a) On the pure aluminum the coating continues to grow until it thickens and cracks due to internal stress; (b) On the 157 alloy, intensified corrosion occurs around the second phase particles, altering the corrosion product, possibly by providing irregularities in the film where stresses are relieved with less damage to the protective nature of the film; (c) On the 155 alloy, corrosion occurs in a manner similar to that on the 157 except that in this case the distribution of irregularities is less uniform and therefore less effective; (d) On the 2S the irregularities in the film are large and far apart and therefore any beneficial effect on the film is small.

Acknowledgement

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Any discussion of this article not published above will appear in June, 1961 issue.

Elevated Temperature Corrosion Of Type 310 Stainless Steel By Vanadium Compounds★

By HUGH L. LOGAN

Abstract

The mechanism of the attack of Type 310 stainless steel by vanadium compounds has been studied. Catastrophic attack by a mixture of 67 wt. percent V_2O_5 + 33 wt. percent of $NaVO_3$ occurred at temperatures of 1900 to 2000 F if the steel had previously been oxidized and air and water vapor were present. In some instances an unidentified phase formed at the steel-slag interface attacked the steel by grain boundary penetration and adsorption of steel grains. A chemical analysis of the slag showed that the chromium-iron and manganese-iron ratios were the same in the slag as in the steel. The nickel-iron ratio, however, was higher in the slag than in the steel. This would suggest the possibility that nickel is selectively absorbed by the slag prior to general attack. 4.2.3, 4.3.3, 3.5.9, 6.2.5

Introduction

AN INVESTIGATION of the corrosion of Type 310 stainless steel in fuel oil ash contaminants has been in progress at the National Bureau of Standards in cooperation with the Bureau of Ships, Department of the Navy. The first task in the investigation was to determine the minimum temperature at which a synthetic oil ash would attack Type 310 stainless steel. The study of the mechanism of this attack on steel was a greater task. Data obtained in the first undertaking and certain data obtained in the second have been reported previously.[1] The present report gives pertinent data obtained since the earlier paper was prepared.

Materials and Procedure

The experimental procedure was essentially the same as that followed in the earlier work. Specimens about 30 inches in length were cut from tubing 0.80 inch in outside diameter with a wall thickness of 0.072 inch. They were from the same lot of steel as that used previously; the composition is given in Table 1. The tubing was seamless and was received in the annealed condition. Specimens were degreased with acetone, pickled for several hours by filling them with cold concentrated nitric acid, and finally thoroughly rinsed and dried. These specimens served as containers in which a synthetic oil ash was heated. One end was tightly packed with glass wool and after the specimen had been partly filled with a synthetic ash mixture the open end was also closed with glass wool. The tube ends were capped so that an atmosphere of wet or dry air(1) or helium could be introduced. In order that specimen temperatures could be accurately determined, thermocouples were spot welded to the outside of the tubes at selected intervals. The specimens were heated in a tube furnace usually in a horizontal position, in the temperature range of 1700 to 2000 F and for periods of 6 to 200 hours.

The synthetic ash mixture used in most of the work to be reported here contained 67 wt. percent of vanadium pentoxide and 33 wt. percent of sodium vanadate (78.7 mol percent V_2O_5 and 21.3 mol percent Na_2O). This mixture contains a little less V_2O_5 than the compound $Na_2O \cdot V_2O_4 \cdot 5V_2O_5$ reported by Shevlin[2] to be the most corrosive of any synthetic ash mixture made up of V_2O_5 and $NaVO_3$. At the minimum temperature of attack of the steel, 1075 F, the mixture would consist of solid $Na_2O \cdot V_2O_4 \cdot 5V_2O_5$ and liquid $NaVO_3$ but at the temperature at which most of the data were obtained the mixture would be entirely molten and highly viscous.[2]

Results and Discussion

Catastrophic failures of several specimen tubes have been obtained. These tubes, prior to being filled with the synthetic ash mixture, were heated in air for a short time at 2000 F in order to produce heavy oxide films on the interior surfaces. Failures of the type shown in Figure 1 were obtained on some but not all of the tubes subsequently filled with the ash mixture and heated with the introduction of a moist air atmosphere at 1900 to 2000 F. No failures were obtained if the atmosphere was dry air or moist helium.

There was severe grooving or pitting of the metal adjacent to the regions where complete failure had occurred (see Figure 2). The attack generally occurred in regions where there had been extensive grain growth. A single grain (except for twin boundaries) extending entirely through what remained of the tube wall in a region that had been severely attacked is shown in Figure 3. The microstructure at the bottom of a small groove, also in the region of severe attack is shown in Figure 4. A tongue of what appeared to be a second phase,(2) most

About the Author

HUGH L. LOGAN has been associated with the National Bureau of Standards since 1936 where his early work dealt with corrosion and stress corrosion cracking of aluminum alloys. He received a BS in chemistry from Tarkio College and an MS in physics from the University of Colorado. He is a member of NACE, ASM, Electrochemical Society and Washington Academy of Sciences.

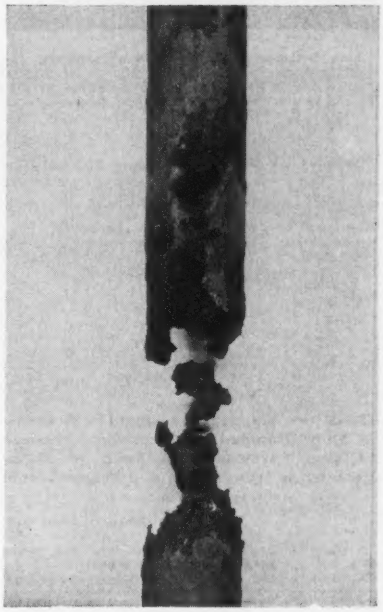

Figure 1—Catastrophic failure of tube specimen. Specimen had been oxidized at 2000 F before introduction of V_2O_5 and $NaVO_3$ mixture. Failure occurred in less than 24 hours at 1950 F, with moist air being introduced into the system. ¾X.

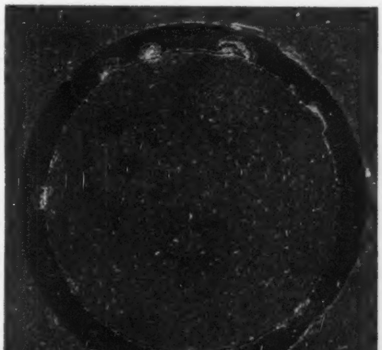

Figure 2—Cross-section of specimen taken adjacent to the area of complete failure shown in Figure 1. Note grooves or pits penetrating well into the interior wall of the specimen. 3X.

★ Submitted for publication November 10, 1960.

(1) Air from a "breathing grade" cylinder was used. This could be moistened by bubbling it through water or dried by passing it through a drying tube containing anhydrous calcium sulfate.

(2) An unidentified phase, unaffected by conventional etchants, was found at the interface between the steel and slag (ash mixture melted and subsequently frozen in contact with the steel) in specimens heated to 1200 F or more. This phase was also found penetrating a short distance into the steel either as a tongue or in a more massive form (see Figure 4 of the previous paper) in some other specimens studied.

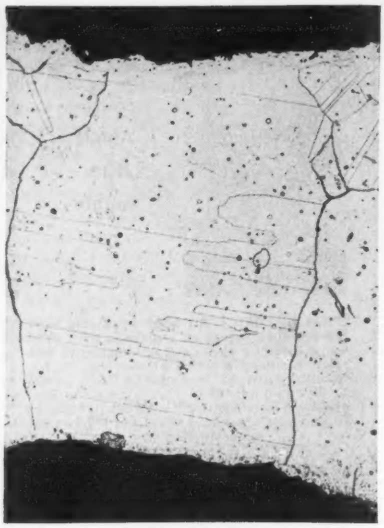

Figure 3—Large grained section of specimen wall that had been reduced to less than ½ its original thickness by the molten V_2O_5-$NaVO_3$ attack at 1950 F. Etched electrolytically in 10 percent oxalic acid, 100X.

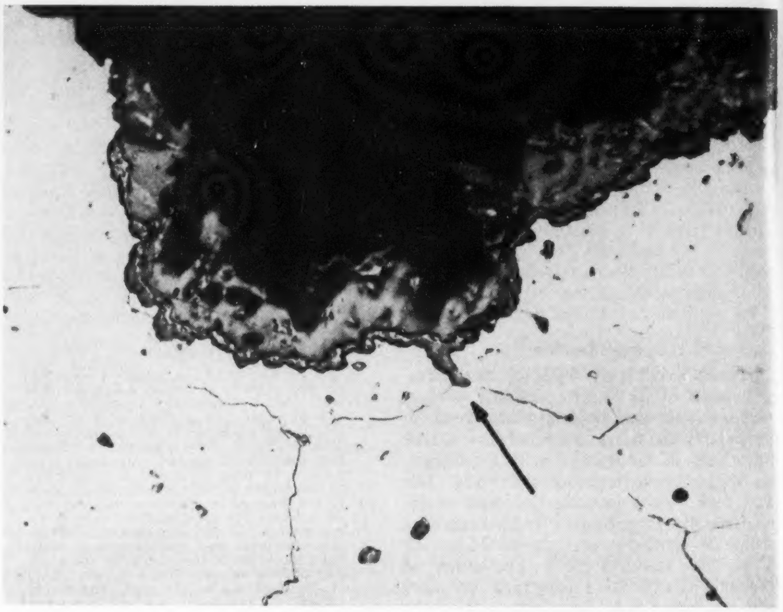

Figure 4—Beginnings of a small groove showing tongue of unidentified phase (marked by arrow) penetrating into the metal along a grain boundary. Specimen heated at approximately 1950 F for about 24 hours. Electrolytically etched in 10 percent oxalic acid, 500X.

TABLE 1—Composition of the Steel Used in the Investigation

Element	Percent
Carbon	0.06
Manganese	1.53
Phosphorus	0.022
Sulfur	0.005
Silicon	0.19
Nickel	21.2
Chromium	24.8

TABLE 2—Composition of Metallic Elements in Slag Obtained in Catastrophic Failure of One Specimen and Ratio of Other Elements to Iron in Slag and Original Steel

		Ratio of Element to Iron	
Element	Composition of Slag, Percent*	In Basis Steel	In Slag
Fe	35.2	1.00	1.00
Cr	16.7	.475	.474
Ni	15.2	.406	.432
Mn	1.04	.029	.030
V	2.6	....	....

* If Fe, Cr, Ni and Mn are considered to be present as simple oxides and V as $Na_2O \cdot V_2O_4 \cdot 5V_2O_5$ the total content of the slag was calculated to be approximately 100 percent.

probably of the same composition as that described in the earlier paper[1] is shown in the area marked by an arrow. This suggests that metal grains may in some cases be surrounded by this phase and subsequently absorbed by the slag. Figure 5 shows another instance where this type of attack was occurring. It was reported in the earlier paper that attack of this type was much more severe in the 302 stainless steel (with a lower alloy content) than in the 310 stainless steel. An example of grains of 302 stainless steel completely surrounded by the second phase was shown in Figure 6 of the earlier paper.

In this connection also, particles of metal that appear to be steel were frequently found in the slag adjacent to the remaining walls of the tubing in specimens that had failed catastrophically; a typical example is shown in Figure 6. It is suggested that these are parts of grains of steel that had been separated from the specimen wall by the second phase and had not yet been completely absorbed by the slag. The lack of general intergranular penetration of the metal to any appreciable depth by a second phase indicates that some of the attack was by other processes. In this connection it was noted that, using certain etchants, grain boundaries were revealed in the interior of the steel but not, except for twin boundaries, in a layer about 0.002 inch thick adjacent to the slag-steel interface. This is shown in Figure 7.

Heating of the $V_2O_5 + NaVO_3$ mixture produced a viscous slag that on solidifying and cooling to room temperature was quite hard. In some instances the slag was divided into two principal layers as is shown in Figure 8. Both layers were very much harder than the steel. The Knoop number of the steel was 175 (range 166-182), that of the layer closest to the steel was 555 (range 506-614), and that of the second layer was 835 (range 740-940).

X-ray, fluorescent and diffraction studies have been made of the slag and spectrochemical determinations and one chemical determination have also been made. In the earlier paper it was reported that $NiO \cdot V_2O_5$, $2NiO \cdot V_2O_5$, $CrVO_4$, $Fe_2O_3 \cdot 2V_2O_5$ and possibly $FeVO_4$ had been identified in the slag. There were usually some unidentifiable lines in the diffraction patterns in which these compounds were identified. It should also be noted that many lines common to several compounds are found in the powder patterns of these materials. In some instances compounds that may actually have been present were ruled out because no lines were found which were particularly characteristic of that compound and that compound alone.

A rather intensive effort was made to separate into its components the slag produced in the catastrophic failure of one of the specimens. The slag was largely ferromagnetic; however, even after it had been finely ground a separation by magnetic methods was not successful. Chemical methods were then used. The solid was leached successively with hot distilled water, sodium hydroxide and oxalic acid solutions and finally hydrochloric acid. Metals present in each of the various residues were identified by qualitative flourescent methods and the compounds by X-ray diffraction methods. This analysis indicated the presence of gamma-Fe_2O_3, Cr_2O_3, NiO, V_2O_3 and a vanadate (not identified) of one or more of the following: chromium, manganese, iron, or nickel. The lattice parameter of the gamma Fe_2O_3 in this slag was 8.326 Å compared with the value of 8.338 recently reported[3] for this oxide. This would suggest that another oxide is in solid solution in the gamma Fe_2O_3. While small particles of the steel were found near the slag-steel interface, Figure 6, X-ray diffraction methods failed to detect any austenitic stainless steel in the slag. This would indicate that the steel particles were combining with either oxygen or V_2O_5 to form oxides or vanadates.

The results of a wet chemical analysis(3) of slag from this same specimen are given, together with the ratios of the

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(3) The slag was not completely soluble in any of the common acids nor aqua regia. It was necessary to fuse it with Na_2O_2 and then leach it with water and acidify in order to get all of it into solution.

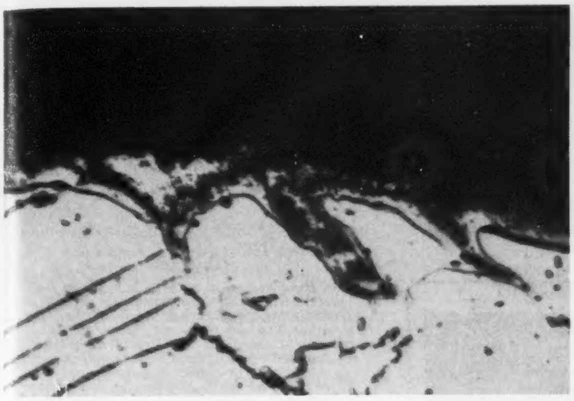

Figure 5—Tongues of second phase penetrating into the steel along the grain boundaries at the slag steel interface and almost completely surrounding one grain of steel. Specimen heated at 1890 F for 6 hours. Etched electrolytically in 10 percent oxalic acid, 750X.

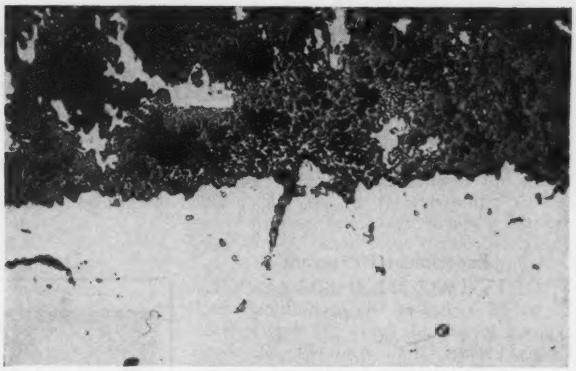

Figure 6—Particles believed to be steel trapped in slag adjacent to the steel that had been heated to approximately 1900 F for about 24 hours. Unetched, 500X.

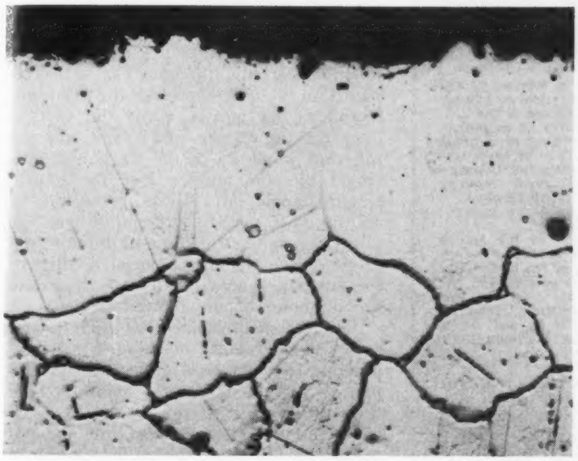

Figure 7—Only twin boundaries were developed adjacent to steel-slag interface in this specimen heated to 1750 F, etched 30 ml HNO_3 + 20 ml CH_3COOH. 500X.

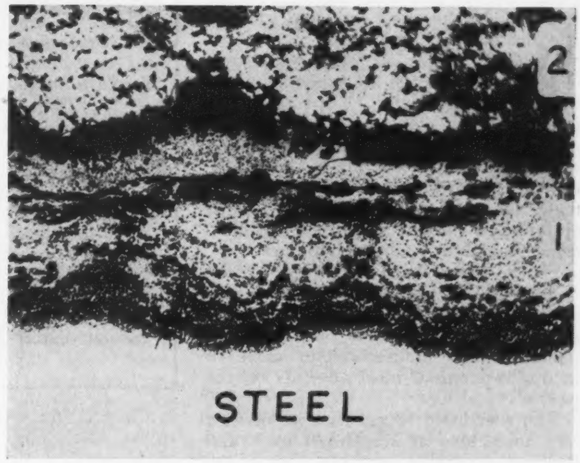

Figure 8—Steel and two layers of slag. Specimen was heated at 2000 F for 24 hours. Steel, layer 1, and layer 2 had average Knoop numbers of 175, 555 and 835 respectively. Unetched, 100X.

various other elements to iron in the slag and original steel, in Table 2. It is interesting to note that while the ratios of both chromium and manganese to iron are very nearly the same in the slag and steel, the nickel to iron ratio is appreciably higher in the slag than in the steel. A semi-quantitive spectrochemical analysis of the slag from another specimen that had not failed also indicated that the nickel-chromium ratio was higher in the slag than in the steel. These data suggest that nickel may be selectively absorbed by the slag prior to general attack. This could explain why the attack on the Type 302 stainless steel (with its lower nickel content) described in the earlier paper,[1] was so much more severe than that on the Type 310 steel used in the investigation.

Summary

1. Catastrophic failures of Type 310 stainless steel specimen tubes have occurred under conditions where moist air was introduced into the tube containing 67 wt. percent V_2O_5 and 33 wt. percent $NaVO_3$ at 1900 to 2000 F, providing the specimen had previously been oxidized.

2. No catastrophic failures occurred in specimens exposed under very similar conditions except that atmospheres of dry air or moist helium were introduced into the tubes instead of moist air.

3. Grooving was found in sections of specimens adjacent to regions where failure had occurred. Grooving was observed in regions where there had been grain growth. In some instances material of a second phase was found at the root of a groove penetrating into the steel along grain boundaries.

4. Using the technique of leaching out various components, the slag produced in a catastrophic failure of a specimen was found, by X-ray fluorescent and diffraction techniques, to contain gamma Fe_2O_3, Cr_2O_3, NiO, V_2O_3 and an unidentified vanadate. The lattice parameter of the Fe_2O_3 was a = 8.326 Å.

5. Chemical analysis of the slag from a tube that had failed catastrophically indicated that the chromium-iron and manganese-iron ratios were the same in the slag as in the basis steel. The nickel-iron ratio in the slag was .432 compared to .406 in the basis steel. This would suggest the possibility that the nickel was selectively absorbed by the slag prior to general attack.

Acknowledgment

This investigation was supported by the Bureau of Ships, Department of the Navy. The author expresses his appreciation to the Bureau of Ships for permission to publish this paper. The conclusions drawn from these data are his own and not necessarily those of the Bureau of Ships.

Joan P. Calvert, June Chung Fugh, James G. Early, Jr., Joseph E. Himes, and Howard T. Yolken assisted in this investigation.

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Investigation of Mild Steel Corrosion Rate In San Diego Harbor★

By M. H. PETERSON and L. J. WALDRON

Abstract

The basic corrosion rate at San Diego has been determined by exposure of an extensive series of mild steel panels for 6, 12, and 18 months. The corrosion rate as determined by weight loss, and the degree of pitting determined by pit depth measurements was measured for unprotected panels, and for three different levels of cathodic protection.

The basic corrosion rate for unprotected panels over the one-year exposure was 25.8 mdd and the greatest pit depth found exceeded 60 thousandths of an inch. The fully protected panels suffered virtually no corrosion while the partially protected panels had a corrosion rate of 8.3 mdd. However, the greatest pit depth found on the partially protected panels was only 9 mils.

6.2.3, 2.2.2, 3.2.2, 5.2.1

Experimental Program

FORTY-TWO MILD steel panels 12 by 12 inches by 1/16 inch thick were exposed for periods up to 552 days in San Diego Harbor. Of these number, 24 were divided into three sets of eight panels each and were used in the primary one-year program. Figure 1 is the test rack used in the primary program. Each rack (one panel set) contained eight panels divided into pairs and mounted back to back. The backs of the panels were painted with rubber-base primer and commercial pipe-wrap tape to confine corrosion to the front face and to insulate the panels electrically.

Cathodic protection of the six protected panels in each set was maintained with galvanic anodes coupled to the panels through a low metallic resistance path. Under these conditions the panels were polarized to essentially the potential of the associated anode. The anode materials used were high purity zinc, aluminum-5 percent zinc alloy, and electrical conductor grade aluminum.

Figure 1 has been simplified by the elimination of the electrical wiring; in addition to the anode-cathode connections, a lead was brought out from each panel through multiconductor cable to facilitate potential measurements on the panels.

The remaining 18 panels were divided into three sets of six panels each and used in a supplementary program to establish a 155-day corrosion rate, a 552-day rate, and to increase the sample size in the determination of the 361-day corrosion rate. The panel racks for this supplementary program contained six panels mounted in pairs back to back on a single spar. No provision was made for potential measurements on these panels.

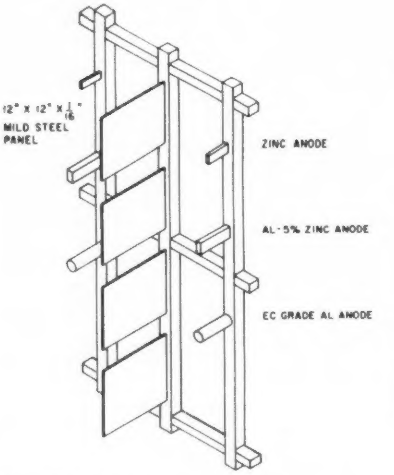

Figure 1—Test rack used for main exposure program.

Peterson

Waldron

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Table 1 is a summary of the panel distribution in the test, and shows the numbers of panels exposed for the various conditions of exposure.

Potential Measurements

The potential of each of the panels in the primary test was measured each week with a Ag/AgCl reference cell and high resistance millivoltmeter. The plotted points on Figure 2 represent the mean potential of six panels, while the vertical lines on the upper graph indicate the range of potential of the six panels protected by zinc anodes. For the unprotected panels, and those protected by aluminum-5 percent zinc alloy and by electrical conductor grade aluminum, the range of the weekly potential reading of the six panels was in all cases less than the diameter of the plotted circle.

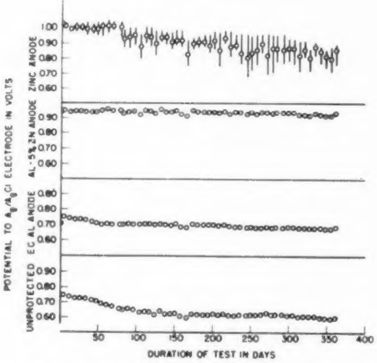

Figure 2—Potentials measured on panels of main test program.

The panels protected by high purity zinc anodes did not maintain a constant potential as had been expected. The six panels protected in this manner held their initial potential of 1.00 to Al/AgCl for about 90 days; at this time the potentials of the panels began to drop in an erratic manner, with the potential of some falling off more rapidly with time than did others.

This drop in potential, which affected all of the panels protected by zinc to some extent, was apparently caused by the build-up of a high resistance film of corrosion products on the zinc anodes. After 361 days all of the zinc anodes were found to be coated with a hard layer of corrosion products with the original saw and punch marks clearly visible on the surface of the coating.

The aluminum-5 percent zinc anodes and electrical conductor grade aluminum anodes maintained reasonably uniform cathode potentials on their associated

TABLE 1—Design of Experiment for Exposure of Mild Steel Panels* in San Diego Harbor

Means of Protection	Number of Racks	Panels Per Rack	Total Panels	Exposure (Days)
Zinc........	3	2	6	361
Al-5% Zn...	3	2	6	361
E.C. Al.....	3	2	6	361
None.......	3	2	6	361
None.......	1	6	6	155
None.......	1	6	6	361
None.......	1	6	6	552

* 12 in. x 12 in. x ¼ in.

★Submitted for publication April 7, 1960. A paper presented at the 16th Annual Conference, National Association of Corrosion Engineers, Dallas, Texas, March 14-18, 1960.

panels for the 361-day exposure period. Over the 361-day period the six steel panels protected by aluminum-5 percent zinc anodes maintained potentials within the range of 0.91 to 0.96 volt to Ag/AgCl. The potentials at the extremes of this range were infrequent; for most of the experimental period the potential variation was much less than the maximum range given.

The potential measurements of the six panels protected by electrical conductor grade aluminum anodes formed a somewhat more complex pattern. For the first 50 days of the test, the potentials measured on the protected panels were identical with those measured on the unprotected panels, and apparently no protective current was supplied by the anodes during this period. During this period, the potentials of both the protected and unprotected panels gradually decreased from an initial value of 0.75 volt, and at the end of the 50-day period the potentials of both measured about 0.72 to Ag/AgCl. For the remainder of the 361-day period, the potential of the protected panels remained within the range of 0.72 to 0.67 volt to Ag/AgCl, while during the same period the potential of the unprotected panels dropped to 0.60 volt.

From these data it is evident that the equilibrium anode potential was in the range of 0.72 to 0.67 volt and that sufficient protective current was supplied by the anodes to maintain the steel cathodes within this potential range for the last seven-eighths of the exposure.

Evaluation Procedure

After exposure, all fouling and loose rust were removed at San Diego prior to shipment of the panels to the U. S. Naval Research Laboratory. The panels were packed in an absorbent material which was inadvertently wetted so that some additional corrosion occurred during shipment. This additional corrosion consisted of surface rust only, and was negligible in all cases with the possible exception of the panels protected by zinc and by aluminum-5 percent zinc.

After cleaning in inhibited hydrochloric acid, the panels were weighed and the weight loss determined by difference from the pre-exposure weights.

TABLE 2—Corrosion of Unprotected Mild Steel Panels in San Diego Harbor—361 Days Exposure

Panel Rack	Weight Loss		Pitting	
	Total (grams)	gm/dm²	Av'g 5 Deepest Pits (mils)	Deepest Pit (mils)
B	78	9	36	47
	80	9	32	38
	74	8	33	40
	85	10	44	49
	85	10	47	56
	81	9	36	47
C	78	9	26	29
	77	9	20	24
D	86	10	53	55
	87	10	56	60
E	77	9	26	30
	85	10	38	42
Mean Value	81	9	37 Median 45	

An estimate of the aggressiveness of the pitting attack was made by dividing the surface of the panels into grids and measuring the thickness of the remaining metal with a needle probe and dial micrometer. The pit depth measurements were repeated for randomly selected panels and yielded reproducible values both for the depth of the deepest pit per panel, and for the depths of the five deepest pits per panel.

Experimental Results for Unprotected Panels

The data for the 361-day exposure of the unprotected panels are shown on Table 2. Treatment of the data by the analysis of variance technique showed no significant difference in the variance of the weight loss between different panels racks as compared to the variance of the same data within racks. These data have been combined to yield a mean weight loss of 9.3 grams per square decimeter with a standard deviation of 0.5 grams per square decimeter. This does not necessarily mean that there was no difference in the weight loss from rack to rack, but that if there were such a difference it was too small to be revealed by an experiment with this sample size.

Pitting does differ significantly from rack to rack, with the deepest and most extensive pitting occuring on rack D. The mean pit depth of the five deepest pits per panel is 37 mils with a standard deviation of 11 mils. The median value of the deepest pit per panel data is 45 mils.

Quantitative interpretation of this pit depth data is extremely difficult as some panels had 12 or more pits deeper than the deepest pits on the less severely attacked panels. One panel had two perforations within the one-year exposure so is safe to assume that the maximum pitting rate at San Diego exceeds 60 mils per year in mild steel.

The authors are unable to account for this great difference in pitting behavior from rack to rack, as all of the racks were hung from a pier at approximately the same depth, and within a total distance along the pier of one hundred feet. A possible explanation is partial shading of some racks from fouling and other influences by pier supports and reserve ships tied to the pier. San Diego was known to have very aggressive water due to heavy contamination of the shallow bay by sewerage; this aggressiveness is apparently increased by the periodic growth of plant organisms in the surfaces of the bay. Under these conditions the surface water layer is supersaturated with oxygen, while the water below this surface layer is aneorobic. This complex situation is further complicated by the mixture of these two water layers under some conditions of tide and wind[1]. The experience of the reserve fleet has been that the current requirements for cathodic protection of reserve ships varies more rapidly and over a wider range than at any other reserve fleet site.

Experimental Results for Protected Panels

The data from the panels protected by high purity zinc anodes are summarized in Table 3. The value of these data are somewhat clouded by the failure of the anodes to maintain the desired panel potential; however, protection was excellent except for the one panel which suffered a weight loss of 0.7 milligram per square decimeter. This was the panel associated with the lower extreme of the range limit lines on the potential graph for these panels.

With this one exception, the panels when removed from the water, although heavily fouled, still had a bright steel surface; it is believed that an appreciable portion of the weight loss reported occurred during shipment. In any event, the mean value of the weight loss was only 0.4 grams per square decimeter, indicating that cathodic protection was virtually complete.

The data from panels protected by aluminum-5 percent zinc anodes are summarized in Table 4. As would be expected from examination of the potential curve for these six panels, the weight losses of these panels are somewhat more uniform than for the previous set. As before, the panels as removed from the water showed bright metallic surfaces under the fouling, and no visible corrosion damage. Also as before considerable surface rust formed during shipment to the U. S. Naval Research Laboratory. The pitting damage to these panels was negligible and the mean weight loss only 0.3 grams per square decimeter, again indicating virtually complete protection from corrosion.

The panel set protected by the electrical grade aluminum anodes yielded the most interesting data set of the experiment (see Table 5). The potential of these panels decreased from an initial value equal to that of unprotected panels to a value of 0.68 volt to Ag/AgCl after 361 days. At this time the unprotected

TABLE 3—Corrosion of Mild Steel Panels Protected by High Purity Zinc Anodes San Diego Harbor—361 Days Exposure

Panel Rack	Weight Loss		Pitting
	Total (grams)	gm/dm²	
C.........	6	0.7	Negligible
	3	0.4	Negligible
D.........	3	0.4	Negligible
	1	0.1	Negligible
E.........	3	0.4	Negligible
	4	0.5	Negligible
Mean Value	4	0.4	....

TABLE 4—Corrosion of Mild Steel Panels Protected by Al-5 Percent Zn Anodes San Diego Harbor—361 Days Exposure

Panel Rack	Weight Loss		Pitting
	Total (grams)	gm/dm²	
C.........	3	0.3	Negligible
	3	0.4	Negligible
D.........	4	0.4	Negligible
	4	0.4	Negligible
E.........	2	0.2	Negligible
	3	0.4	Negligible
Mean Value	3	0.3	....

TABLE 5—Corrosion of Mild Steel Panels Protected by EC Grade Aluminum Anodes San Diego Harbor—361 Days Exposure

Panel Rack	Weight Loss		Pitting	
	Total (grams)	gm/dm²	Av'g 5 Deepest Pits (mils)	Deepest Pit (mils)
C	35	4	3	5
	24	3	3	4
D	24	3	4	6
	22	3	8	9
E	26	3	3	5
	26	3	3	3
Mean Value	26	3	4	Median 5

TABLE 6—Corrosion of Unprotected Mild Steel Panels in San Diego Harbor—155 Days Exposure

Panel Rack	Weight Loss		Pitting	
	Total (grams)	gm/dm²	Av'g 5 Deepest Pits (mils)	Deepest Pi (mils)
A	51	6	17	19
	50	6	19	20
	44	5	13	19
	48	6	18	18
	47	5	24	30
	45	5	19	25
Mean Value	47	5	18	Median 20

TABLE 7—Corrosion of Unprotected Mild Steel Panels in San Diego Harbor—552 Days Exposure

Panel Rack	Weight Loss		Pitting	
	Total (grams)	gm/dm²	Av'g 5 Deepest Pits (mils)	Deepest Pit (mils)
F	114	13	32	38
	115	13	35	45
	128	15	43	49
	116	13	24	26
	115	13	38	45
	123	14	41	50
Mean Value	118	14	35	Median 45

panels had a potential of 0.61 volt indicating that the protected panels were polarized by about 70 millivolts. During the first 50 days of the test, the solution potential of the unprotected mild steel was apparently more anodic than that of the E.C. grade aluminum material and no current flowed to the steel plates. From 50 days throughout the remainder of the test the solution potential of the panels was low enough so that some current flowed to the panels and slight polarization was maintained.

This slight polarization, which reached a maximum value of 70 millivolts after 361 days, was sufficient to reduce the weight loss from the 9 grams per square decimeter suffered by the unprotected panels to 3 grams per square decimeter. The effect on the pitting rate was even greater; pitting as such was virtually eliminated and the corrosion attack on the panels was of a general nature. The mean of the pit depths of the five deepest pits per panel was reduced from 37 mils for the unprotected panels to 4 mils. The deepest pit found on any of the six panels was only 9 mils. This pit occurred on one of the panels in rack D; pitting of the unprotected panels was also at a maximum on this rack with a pit depth exceeding 60 mils (perforation). Thus on this rack, the pitting rate was reduced from greater than 60 mils per year to 9 mils per year. This is convincing evidence that, at least for mild steel, the application of cathodic protection at levels well below the optimum polarization level is effective both in reducing general corrosion and in eliminating severe pitting.

Supplementary Data

Table 6 lists the data for 155 days exposure. The weight loss for this exposure was 5 grams per square decimeter and the mean value for the five deepest pits per panel was 18 mils.

Table 7 shows the same data for 552 days exposure. Here the weight loss was 14 grams per square decimeter and the mean pit depth 35 mils.

Conclusions

It has been established that the use of galvanic anodes coupled directly to experimental panels through a low resistance circuit provides a reliable method for maintaining constant potential control of cathodically protected panels. In addition, the corrosion rate in San Diego Harbor has been established as 9 grams per square decimeter for 361-days exposure, and the maximum pitting rate is greater than 60 mils per year.

Polarization to 0.95 volt to Ag/AgCl at this site gave virtually complete protection from corrosion. Polarization of as little as 70 millivolts reduced the weight loss by two thirds and reduced the maximum pit depth by an even greater factor.

Acknowledgment

This investigation was sponsored by the Bureau of Ships and is part of a study of cathodic protection as applied to the ships of the San Diego Group, Pacific Reserve Fleet.

Reference

1. E. G. Corcoran and J. S. Kittredge. *Corrosion Prevention and Control*, 3, No. 12, 45 (1956).

DISCUSSION

Question by F. W. Fink, Battelle Memorial Institute, Columbus, Ohio:

Did cathodic protection affect the degree of fouling?

Reply by M. H. Peterson and L. J. Waldron:

The authors have never observed any differences in fouling attributable to cathodic protection, except in the immediate vicinity of impressed current anodes where there has been considerable release of chlorine.

Question by Thomas J. Lennox, Jr., South Plainfield, New Jersey:

What were the area relationships between anodes and steel plate cathodes in your test?

Reply by M. H. Peterson and L. J. Waldron:

The high purity zinc anodes had a surface area of 21 square inches, the aluminum-5 percent zinc anodes an area of 31½ square inches, and the EC grade aluminum anodes an area of 41 square inches.

Comment by Thomas J. Lennox, Jr. relative to above reply:

Perhaps the difference in anode sizes can explain in part the higher cathode potentials observed on the aluminum anode protected plate compared to the zinc anode protected plate, since one would expect more instantaneous current output from a long thin anode. In addition, the aluminum anode used had approximately 45 percent more surface area than the zinc anode.

Reply by M. H. Peterson and L. J. Waldron:

While Mr. Lennox' comments about the effect of area and shape on the current out-put of anodes are true in general, I do not think that they are the explanation in this case. All of the anodes used had sufficient instantaneous current out-put to polarize the associated cathodes immediately. In fact, the zinc anodes polarized their cathodes to 1.00 volt to Ag/AgCl and maintained these potentials for 90 days before the potential began to fall. This initial high level of polarization plus the hard deposit found on the zinc anodes and the lack of deposit on the aluminum-5 percent zinc anodes makes me confident that these corrosion products were the cause of the reduced current output and decreased polarization.

Behavior of Stainless Steels and Other Engineering Alloys In Hot Ammonia Atmospheres*

By *J. J. MORAN, J. R. MIHALISIN* and *E. N. SKINNER*

Abstract

Eighteen alloys were tested in hot ammonia atmospheres over long periods of time and results compared. Specific comparison was made of tests made in an ammonia converter and in a chemical plant ammonia line. Materials tested include 11 steels, 37 Ni/20 Cr, 60 Ni/15 Cr, 80 Ni/20 Cr, Inconel, Monel and 99.49 percent pure nickel. It was found that in several ammonia-bearing atmospheres of widely-varying nitride potential, the nickel-base alloys in the iron-nickel-chromium system were substantially more resistant to attack by nitrogen than those alloys richer in iron and chromium.

4.3.3, 6.3.10

Introduction

THE USES OF ammonia are many and diverse. The production of munitions, textiles, fertilizers, rubber products, and petroleum products among others consumes vast quantities of this product annually. Ammonia is important metallurgically, both in winning metals from their ores and in providing a wear-resistant surface on steels by "nitriding." Also, it is the most commonly used refrigerant, particularly for large industrial installations. In the vicinity of four million tons of synthetic anhydrous ammonia are produced each year.

Although this enormous quantity is produced, sometimes transferred through tubes and, in certain applications, used at elevated temperatures, little quantitative information is available on the behavior of metals in hot ammonia atmospheres. Vanick[1] has described the formation of a brittle, non-metallic, iron nitride layer on steels exposed to decomposing ammonia at temperatures above 400 C and notes that its destructive action is at a maximum between 550 and 700 C. In a subsequent paper[2] he reported on the exposure of a variety of alloys to "a synthesizing ammonia-gas mixture at a temperature of 500 C and a pressure of 100 atmospheres." Many of the specimens suffered intergranular fissuring but as no nitrides were observed metallographically it was concluded that the compounds are unstable in this environment and do not contribute directly to the deterioration. Nelson[3] on the other hand, observed the formation of a thin, hard, brittle, nitride layer on the surface of ferrous materials in an ammonia plant operating at 850 F and 2140 psi. Ihrig[4] inserted samples of low alloy, straight chromium stainless, and austenitic stainless steels in the catalyst bed of an ammonia synthesis plant where they were subjected to a pressure of 13,000 to 15,000 psi and a temperature range of 480 to 593 C for 1788 hours. He reported that the austenitic chromium-nickel steels had the thinnest cases of any studied.

Jones[5] conducted an extensive investigation of the nitriding characteristics of a wide variety of steels, ranging from low alloy to austenitic stainless and showed that the latter were the most resistant. Otherwise, in connection with the nitriding of steels (except for ascertaining which heat-resistant container alloys will not impair the efficiency of the process by causing premature dissociation of the ammonia), little consideration has been given to the effect on high-alloy materials of prolonged exposure to the hot gas.

This paper will cover the results obtained from a high temperature test rack, containing 18 alloys, installed within the catalyst basket of an ammonia converter for a period exceeding 29,000 hours (more than three years). For comparison, data from a similar test rack installation in a chemical-plant ammonia line will be presented. The second exposure was in a considerably more aggressive environment, for 1540 hours (2 months). As will be noted presently, the temperature in both cases was approximately the same although in most other respects the conditions differed materially. Over a number of years other test racks have been exposed in different ammonia-containing atmospheres. Some of these data will be included to show the pattern of behavior of the several alloy types under varying conditions which have in common the ability to cause nitriding.

Ammonia Converter

In practically all modern plants one of the several modifications of the original Haber-Bosch process is used to synthesize ammonia. Ample descriptions are contained in the literature and it will suffice here to note the process consists essentially in preparing a three to one mixture of hydrogen and nitrogen and synthesizing to ammonia under pressure in the presence of a suitable catalyst. Depending upon the modification, the

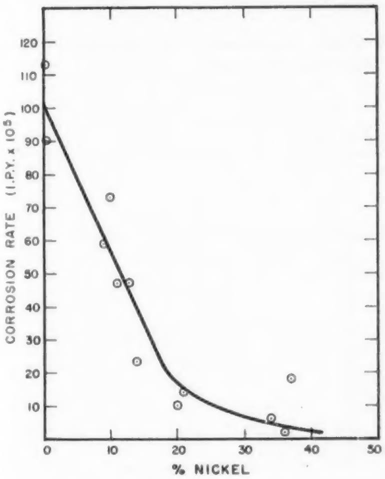

Figure 1—Corrosion behavior of nickel alloys in ammonia converter.

TABLE 1—Chemical Composition of Materials Comprising Test Rack from Ammonia Converter

Material	ELEMENT, PERCENT						Others
	C	Mn	Si	Cr	Fe	Ni	
Type 446	.08	.45	.34	24.82	Bal.	.16	
Type 430	.08	.40	.36	16.75	Bal.	.27	
Type 302B	.10	1.80	2.54	17.74	Bal.	9.50	
Type 304	.07	.94	.39	18.75	Bal.	9.35	
Type 316	.05	1.73	.34	17.25	Bal.	12.98	2.23 Mo
Type 321	.05	1.36	.49	17.60	Bal.	10.63	.52 Ti
Type 309	.13	1.88	.25	22.70	Bal.	14.38	
Type 310	.09	1.80	.38	24.80	Bal.	20.85	
37 Ni/20 Cr	.06	.92	.34	20.21	43.63	37.73	
Type 314	.11	1.88	2.25	24.82	Bal.	19.21	
Type 330 (a)	.07	1.09	.47	15.50	Bal.	34.35	
Type 330 (b)	.14	.80	1.00	14.77	Bal.	36.18	
80 Ni/20 Cr	.06	1.97	1.46	20.43	.38	Bal.	
60 Ni/15 Cr	.05	.09	1.28	15.82	Bal.	58.51	
Inconel Alloy*	.04	.20	.15	14.50	6.31	78.67	
70 Ni/20 Cr	.03	.24	.24	19.21	9.03	71.12	
Monel Alloy*	.12	.95	.14	......	1.33	65.81	31.62 Cu
Nickel	.03	.25	.02	......	.11	99.49	

* Trademark of The International Nickel Company, Inc.

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Skinner

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J. R. MIHALISIN is a senior metallurgist with The International Nickel Company, Inc., Research Laboratory, Bayonne, N. J. He served in U. S. Army from 1943-1945, returning to Yale University where he received a BE in Metallurgy in 1949. In 1953 he received an Sc.D in Metallurgy from MIT. From 1953 to 1955 Dr. Mihalisin was associated with Curtiss-Wright Corporation as a metallurgical supervisor. He is a member of Tau Beta Pi, Sigma Xi, AIME, ASM, and EMSA.

temperature may vary from 400 to 650 C and the pressure from 1,500 to 15,000 psi.[8]

In the present test the temperature in the converter was 490-550 C (915-1024F), the average pressure 5,200 psi and, as mentioned above, the duration of the test 29,164 hours—approximately $3\frac{1}{3}$ years. The test rack was installed within a catalyst basket and the concentration of ammonia at this point was reported as 5-6 percent. The gas produced was of roughly the following proportion by volume: hydrogen, 60; nitrogen, 20; argon, 8; and ammonia, 12. Table 1 lists the compositions of the alloys contained in the test rack.

Upon removal from the converter the rack was dismantled and the specimens were sectioned and prepared for metallographic examination to determine the total depth of altered metal or case. From these measurements the rates of attack have been calculated, and are shown in Table 2 and plotted in Figure 1. Photomicrographs, representative of the altered structures observed in the several alloy types are shown in Figure 2. The structures were also examined by x-ray diffraction to identify the constituents present. These results are shown in Table 3. There is no question that the highest rate of attack was experienced by the ferritic alloys, less by the austenitic iron-nickel-chromium stainless steels and little or none by the high nickel-chromium or nickel-copper alloys. Actually the alloys containing more than 30 percent nickel, with a single exception, did not form a nitride phase although the presence of nitrogen in Type 330 is indicated by the expanded lattice and by the etching behavior of the surface layers.

Plant Ammonia Line

The test rack containing the alloys listed in Table 4 was placed in a 3-inch diameter preheater exit line where the gas temperature was approximately 500 C (935 F) and remained in this location for the duration of the exposure, 1540 hours. Duplicate samples of each material were exposed. One set was cleaned and polished and the other preoxidized at 1800 F for 15 minutes and air-cooled. The gas pressure was not reported, but the velocity was 30 feet per second and it was known that considerable turbulence existed within the line both from the presence of the rack and because the line made a right angle turn immediately downstream from the rack. The composition of the gas was given as 99.1 percent ammonia and 0.9 percent inerts, presumably products of dissociation. However it was noted that the samples, upon removal from the line, were coated with carbonaceous deposit which may have originated either from thermal cracking of small amounts of hydrocarbons or carbon monoxide in the gas or from a back-diffusion of carbon from the vaporizer during a previous shut-down.

When the rack was removed at the conclusion of the test it was clearly evident that extensive corrosion attack had occurred on a number of the alloys and in fact the Type 316 specimen had suffered nearly complete disintegration.

The thickness of each specimen had been measured prior to the test and was measured again at the same locations at the conclusion. The specimens were then examined metallographically to measure the depth of penetration. The extent of corrosion, referred to the original thickness, is reported in Table 5 and plotted in Figure 3.

Representative photomicrographs are shown in Figure 4. It will be noted that some of the samples contain two layers of corrosion product; one, the outer, dark etching, obviously brittle, and nonmetallic in appearance; the other, lighter etching, uncracked and obviously an integral part of the unaffected metal beneath it. The x-ray diffraction data, obtained from the converter-test samples discussed previously, show that the outer layer is largely the intermetallic compound CrN whereas the inner consists of nitrogen in solid solution in the matrix. Again it will be observed that the high

TABLE 2—Corrosion Behavior in Ammonia Converter*

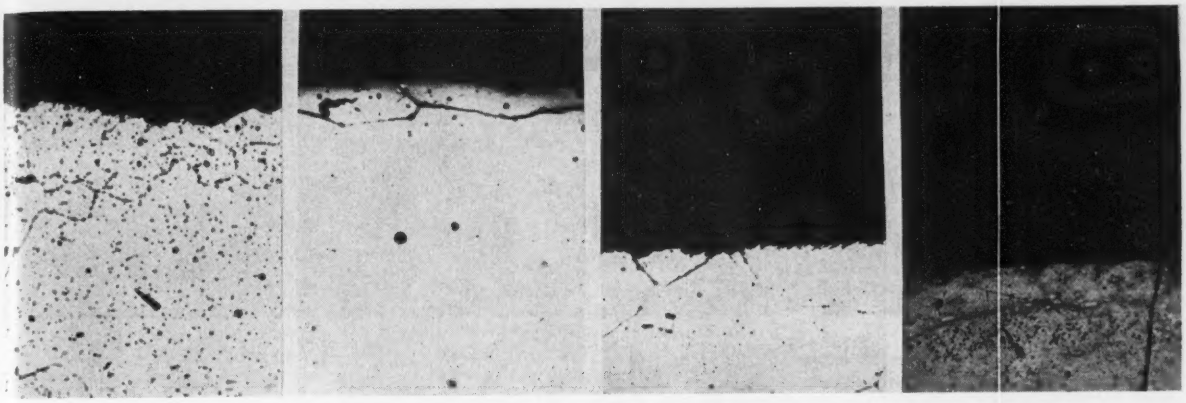
Alloy	Nominal Nickel Content, Percent	Corrosion Rate (ipy)
446	..	0.00112
430	..	0.00090
302B	10	0.00073
304	9	0.00059
316	13	0.00047
321	11	0.00047
309	14	0.00023
310	21	0.00014
37 Ni/20 Cr	37	0.00018
314	20	0.00010
330 (a)	34	0.00006
330 (b)	36	0.00002
80 Ni/20 Cr	80	......
60 Ni/15 Cr	60	......
Inconel Alloy	79	......
70 Ni/20 Cr	70	......
Monel Alloy	66	......
Nickel	99.5	......

* Based on 29,164 hours exposure.

TABLE 3—Results of X-Ray Examination of Specimens from Ammonia Converter

Alloy	Nominal Nickel Content, Percent	STRUCTURE	
		Surface Layer	Base Metal*
446	..	γ—Cr N	BCC
430	..	γ—Cr N	BCC
302B	10	γ—Cr N, BCC	FCC
304	9	γ—Cr N, BCC	FCC
316	13	γ—Cr N, BCC	FCC
321	11	γ—Cr N, BCC	FCC
309	14	γ—Cr N, BCC	FCC
310	21	γ—Cr N, Weak BCC	FCC
37 Ni/20 Cr	37	γ—Cr N	FCC
314	20	γ—Cr N, Weak BCC	FCC
330 (a)	34	Expanded FCC	FCC
330 (b)	36	Expanded FCC	FCC
80 Ni/20 Cr	80	FCC	FCC
60 Ni/15 Cr	60	FCC	FCC
Inconel Alloy	79	FCC	FCC
70 Ni/20 Cr	70	FCC	FCC
Monel Alloy	66	FCC	FCC
Nickel	99.5	FCC	FCC

* BCC = Body-Centered Cubic.
FCC = Face-Centered Cubic.



80 Ni-20 Cr
No loss

35 Ni-15 Cr
0.00002

8 Ni-18 Cr
0.00059

0 Ni-28 Cr
0.00112

Figure 2—High temperature corrosion of nickel alloys by ammonia synthesis gas. Exposure time: 29,164 hours at 490-550 C. Corrosion losses (in ipy) are given directly under the alloys.

nickel alloys, although not pure nickel itself, are more resistant than the alloys higher in iron and chromium. It will also be noted that some improvement was realized by the oxidation treatment which imposed a comparatively inert barrier between the active gas and the underlying metal.

Comparison of Behavior

Data have been presented above on the behavior of a number of commercial alloys when exposed to two dissimilar ammonia-containing atmospheres. For comparison these data have been plotted together with the results from two other test rack exposures. (See Figure 5).

The exposure conditions in the ammonia heater (Curve C) were as follows: the feed was a 20:1 mole ratio of ammonia to an organic gas, the temperature was 460 C (861 F), the rack was subjected to 12 startups and shutdowns, and the time on stream was 939 hours (actually 1011 hours of operation including startups and shutdowns). The samples in the nitriding furnace (Curve D) were exposed for 586 hours to an unspecified number of two-stage nitriding cycles. The first stage was carried out at 524 C (975 F) and 20 percent ammonia dissociation and the second at 565 C (1050 F) and 85 percent dissociation. The time for each stage in a given cycle is a function of the case depth desired on the work and may vary from

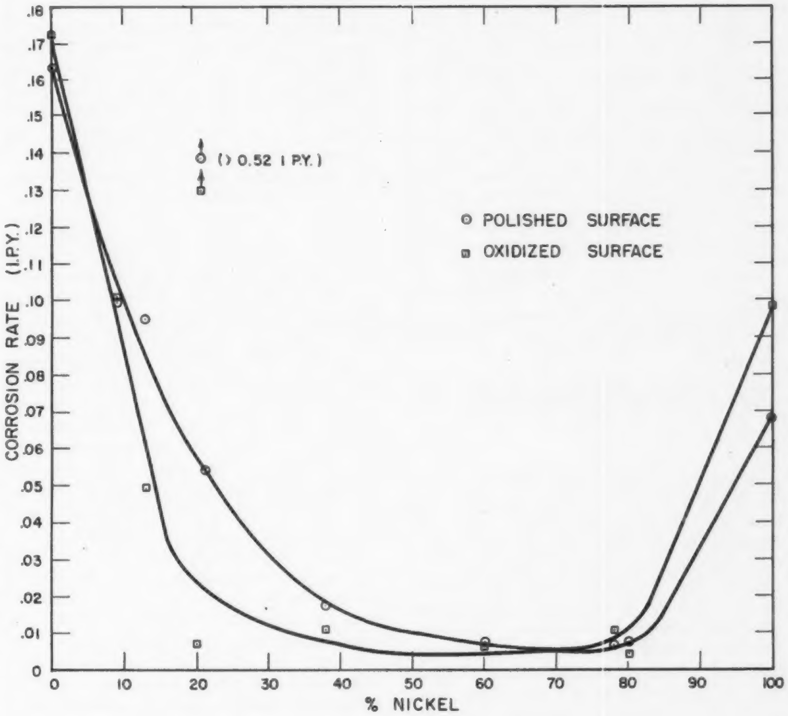
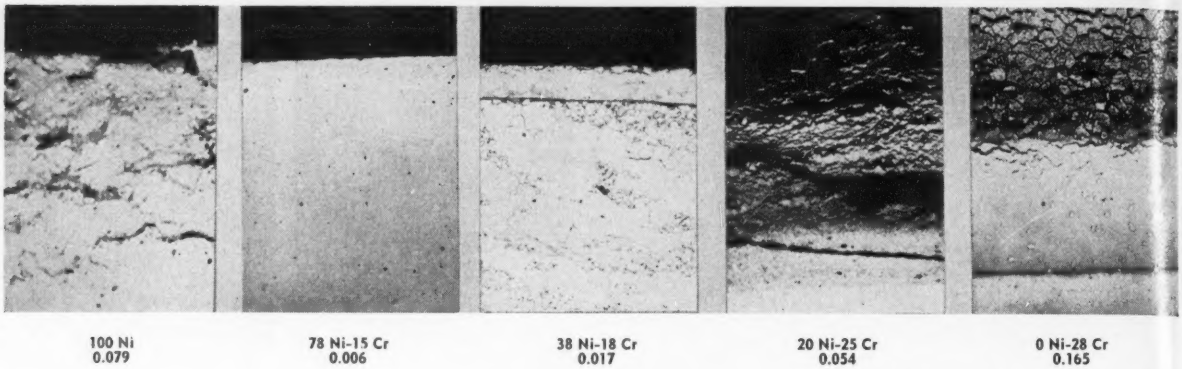


Figure 3—Corrosion behavior or surface polished and surface oxidized nickel alloys in plant ammonia line.

TABLE 4—Chemical Composition of Materials Comprising Test Rack from Plant Ammonia Line

Alloy Type	ELEMENT, PERCENT							
	C	Si	Mn	S	Cr	Ni	Fe	Other
Nickel	.03	.01	.22	.005	....	99.46	.17	.08 Cu
80 Ni/20 Cr	....	1.3	....	....	19.5	Bal.	....	(Nominal)
Inconel Alloy	.05	.25	.19	.007	14.07	78.48	6.81	
60 Ni/15 Cr	...	1.3	....	....	15	60	Bal.	(Nominal)
38 Ni/18 Cr	....	1.3	....	....	18	38	Bal.	(Nominal)
Type 310	.127	1.0	1.97	.010	24.07	21.63	Bal.	
Type 309	.093	.23	1.82	.012	23.58	13.26	Bal.	
Type 316	.077	.46	1.71	.020	17.39	12.30	Bal.	2.40 Mo
Type 304	.055	.60	1.32	.019	18.69	9.28	Bal.	
Type 446	.20	.38	.54	.013	28.52	....	Bal.	


100 Ni
0.079

78 Ni-15 Cr
0.006

38 Ni-18 Cr
0.017

20 Ni-25 Cr
0.054

0 Ni-28 Cr
0.165

Figure 4—High temperature corrosion of alloys by anhydrous ammonia. Exposure: 1540 hours at 500 C. Corrosion losses of alloys are reported in IPY's directly underneath the respective alloys.

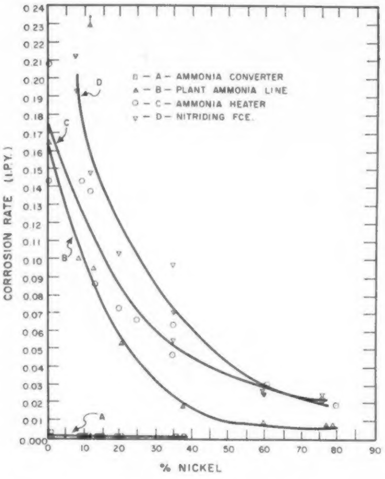


Figure 5—Corrosion behavior of commercial alloys exposed to dissimilar ammonia-containing atmospheres.

TABLE 5—Corrosion Behavior in Plant Ammonia Line*

Alloy	Nominal Nickel Content, Percent	CORROSION RATE, (ipy)	
		Surface Polished	Surface Oxidized
Nickel	100	0.0790	0.098
80 Ni/20 Cr	80	0.0074	0.004
Inconel Alloy	78	0.0063	0.010
60 Ni/15 Cr	60	0.0074	0.006
38 Ni/18 Cr	38	0.0171	0.010
Type 310	21	0.0535	0.007
Type 309	13	0.0950	0.049
Type 316	12	>0.52	>0.5
Type 304	9	0.0995	0.102
Type 446	..	0.1645	0.173

* Based on 1540 Hrs' Exposure at 500 C.

TABLE 6—Alloys Included in Tests

Ammonia Heater	Nitriding Furnace
Nickel	Inconel Alloy
80 Ni/20 Cr	60 Ni/15 Cr (2 Samples)
Inconel Alloy	Type 330 (2 Samples)
60 Ni/15 Cr	35 Ni/15 Cr (Cast)
35 Ni/15 Cr	Incoloy Alloy
Incoloy Alloy*	Type 310
Type 311	Type 309
Type 310	Type 302B
Type 309	Type 302
Type 316	
Type 304	
Type 410	
Type 446	

* Trademark of The International Nickel Company, Inc.

5 to 8 hours for the first and from 0 to 42 hours for the second. No cycles of less than 25 hours total time were run during this test. The alloys contained in each rack are indicated in Table 6.

The data are scattered as plotted because, although it is clearly evident that with increasing nickel content the tendency to nitride is reduced, no attempt has been made to evaluate the contribution of the elements present other than nickel. It is obvious, however, from the data that of two alloys having the same nickel content, but varying in chromium and iron, the one containing the greater quantity of chromium will nitride more (ignoring the effects of minor constituents) because chromium reacts more avidly with nitrogen than does iron. It was not considered worthwhile to study this effect in more detail because the service conditions in field tests of this type are not known with sufficient precision to permit accurate quantitative analysis.

Discussion

The data reported here in general support past experience that the high nickel alloys, although not pure nickel, are inherently better suited for service under nitriding conditions. This is not intended to imply that resistance to nitriding is of primary concern in all of the environments considered; obviously the rate of attack in the ammonia converter was very low and susceptibility to hydrogen embrittlement or attack is of considerably more importance in the selection of materials of construction for this service. Nevertheless it is of interest to note that the behavior of the alloys, with few exceptions, is consistent throughout despite the wide variation of nitriding potential between the several atmospheres.

The extraordinary behavior of Type 316 in the ammonia line test (B) should be commented upon if only to indicate that no satisfactory explanation can be offered. As has been noted above, the presence of additional nitride-forming elements, including molybdenum, in a given base should increase the nitriding tendency of the alloy. It has been generally borne out in examinations of service failures that Type 316 is not notable for its resistance to nitriding. Nevertheless, catastrophic failures had not been previously observed and in fact the Type 316 specimen in the ammonia heater test (C) behaved in a similar fashion to or slightly better than Type 304. Perhaps the molybdenum contributed to the development of an unusually friable or exfoliating scale which spalled excessively under the action of the turbulent gas stream. Unfortunately, not enough of the Type 316 specimens remained to permit any detailed investigation.

The behavior of commercially pure nickel in several of these tests also appear anomalous in view of the demonstrated benefits derived from its presence in combination with other elements. Nickel does not react with nitrogen gas at least up to 900 C[7] and the solubility of nitrogen in nickel, detectable by x-ray methods, is small. (It has been estimated at 0.07 percent by weight.[8]) By treating fine nickel powder with ammonia at 500-550 C several investigators have obtained preparations containing 7.5 percent[8, 9] to 9 percent[10] of nitrogen. X-ray analysis has established the existence of Ni_3N (7.37 percent N) possessing a hexagonal close-packed structure of nickel with interstitial nitrogen atoms.[8] This compound decomposes above 360 C in an evacuated vessel.[8] Thus it appears possible to form a nickel nitride in the temperature range of the tests reported here. In fact, in view of the thermal instability of the compound it is likely that the compound will form only under conditions very similar to these.

Although nickel nitride could, theoretically, form, x-ray diffraction analysis of the commercially pure nickel sample in the ammonia heater test (C) showed only the presence of manganese nitride and graphite in the nickel matrix. "A" nickel contains up to 0.35 percent manganese, and it is concluded that nickel in massive form will nitride only with great reluctance if at all. It is doubtful, moreover because of the low solubility of nitrogen in nickel, that nitride formation would have progressed very rapidly unless some other reaction opened a path to the interior of the sample. That is, if the rate were dependent upon diffusion of nitrogen into the nickel the reaction rate would have been quite low. However, in tests B and C, where substantial penetration of the nickel samples was observed, carbonaceous materials were also present and metallographic as well as diffraction evidence of grain-boundary graphitization was seen. It appears then that the nickel samples absorbed carbon from the atmosphere and precipitated it as grain boundary graphite. The grain boundary discontinuities formed in this fashion could provide an easy path for the comparatively rapid migration of nitrogen to the interior of the sample. In test A where carbon was not present the nickel was apparently unaffected by the atmosphere; in test C where organics were present, rather deep grain boundary attack was observed and indications of nitrides were found; in test B, both graphite and nitride were found in close proximity.

When combined with elements such as chromium and iron that readily form nitrides, the important contribution of nickel is undoubtedly related to its effect on the solubility and hence the diffusion rate of nitrogen in the alloy. Abrahamson and Grant[11] have shown that at 800 C in excess of 1.0 percent nitrogen will go into solid solution in a 50 nickel-50 chromium alloy whereas this is reduced to aproximately 0.1 percent in an 80 nickel-20 chromium alloy. Muhlberg[12] studied the iron-nickel-nitrogen system and found that "the absorption of nitrogen diminishes steeply with increasing nickel content." Muhlberg also noted that for alloys containing more than roughly 30 percent nickel only two phases exist; one a FCC (face-centered cubic) γ, iron-nickel solid solution with dissolved nitrogen and the second a FCC nitride phase. In alloys containing less than about 30 percent nickel, in the vicinity of 500 C, the two FCC phases are accompanied by a BCC (body-centered cubic) α, iron-nickel solid solution containing dissolved nitrogen. Interestingly, as shown in Table 3 only those alloys having nickel contents less than 30 percent of the total of iron plus nickel, in the ammonia converter test, contain a BCC phase. Presumably during the nitriding of iron-nickel-chromium alloys, the chromium because of its greater reactivity with nitrogen, is preferentially consumed leaving at the surface a matrix close enough to an iron-nickel alloy containing dissolved nitrogen to conform to Muhlberg's findings.

Conclusion

It has been shown that in several ammonia-bearing atmospheres of widely-varying nitriding potential the nickel-base alloys in the iron-nickel-chromium system are substantially more resistant to attack by nitrogen than those alloys richer in iron and chromium. It has been suggested that nickel, when in combination with more strongly nitride-forming elements, acts primarily to reduce the solubility and hence the diffusion rate of nitrogen in the alloys resulting in a marked reduction in the rate of corrosive attack. Commercially pure nickel on the other hand is not necessarily immune to reaction with ammonia and the extent of attack may be increased by other contaminants in the gas stream.

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Any discussion of this article not published above will appear in June, 1961 issue.

ERRATUM

Glass Linings and Vitreous Enamels—A Report of NACE Technical Unit Committee T-6H. Corrosion, Vol. 16, 615t-621t (1960) Dec.

On Page 621t, Line 3 of "Acknowledgment" should be amended to read:

pany, the Glascote Company, and Vitreous Steel Products Company for their

Intergranular Corrosion Of Commercially Pure Zirconium★

By *B. S. PAYNE and D. K. PRIEST*

Abstract

Selective corrosion in a hot hydrochloric acid service was noted in a narrow band of the heat affected zone of a commercially fabricated zirconium weldment. Little attack was present on the weld proper or the parent stock. The selective attack has been found to be intergranular in nature and associated with the presence of a precipitated phase in the grain boundaries. The particular precipitated phase required for this attack has been duplicated in bulk samples by specific heat treatment, as well as in experimental weldments.

Two methods of prevention of this localized attack near welds have been evaluated; namely: (1) Use of high purity zirconium, and (2) heating of commercially pure zirconium weldment at 1800 F followed by a water quench. Possible mechanisms for the microstructural changes and for the corrosion attack are discussed.

6.3.20, 3.2.2, 4.3.2, 3.7.3

Priest

Payne

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Introduction

THE USEFULNESS of zirconium in hydrochloric acid environments has been well established in laboratory studies by Golden[1,2,3,4,5], Kuhn[6] and others. In addition, the metal's performance in actual process environments of hydrochloric acid has been judged excellent within permissible ranges of temperature and acid concentration as defined by laboratory test data. It was of considerable interest, in view of this accepted background information to encounter, recently, a localized "weld decay" type of corrosion failure in a welded zirconium tube reportedly handling a 15 percent HCl-85 percent organics solution at 240 F.

The nature of the attack, apparently associated with welding practice, and the lack of extensive field experience on the behavior of zirconium in hydrochloric acid service, prompted the writers to undertake an investigation of the cause of this type of failure.

Procedure

The weldment involved was a 2 inch diameter, 24 inch long welded zirconium pipe liner 0.065 inch in thickness. After sectioning, macro-photographs were made of the pipe interior to show the nature and extent of the attack. Samples were cut to reveal weld cross sections. These were mounted, polished with Linde B 5125 aluminum oxide on a Buehler AB microcloth and etched-polished on the wheel using a solution of 45 parts water, 45 parts concentrated nitric acid and 15 parts of 48 percent hydrofluoric acid.

In the course of the investigation the importance of metal composition was established. A major part of the work was concerned with two heats of zirconium, a commercially pure material (Ingot CP-2) and a high purity material (Ingot 2984). A detailed analysis of these heats is given in Table 1. The analysis data also indicate that the effect of hydrogen content on the occurrence of the localized corrosion was investigated. These hydrogen levels were established for a given material by employing hydrogen charging techniques or vacuum annealing.

Initial experimental work was aimed toward reproducing the localized weld-associated failure in additional zirconium material. Accordingly, test weldments from Ingot CP-2 and Ingot 2984 were made. Typical samples were examined metallographically.

The localized nature of the corrosion attack (adjacent to a weld) and the transient time-temperature effects associated with weld heat affected zones suggested that an extended series of heat treatments would be required to establish the microstructure susceptible to attack. Therefore, both commercially pure and high purity samples were heated in argon at temperatures of 1500, 1600, 1700, 1800 and 1900 F for 5, 10, 20, 30 and 40 minutes and then quenched in water or slow cooled at 10 F per minute to a temperature (1000 F) well below the alpha-beta transformation. Each heat treated sample was examined metallographically. Photomicrographs were taken of the majority of samples. The test weldments were subjected to several corrosion tests. The environment in which the zirconium pipe liner failed had been reported initially as 15 weight percent of 28 weight percent hydrochloride acid and 85 percent of non-corrosive organic materials at a temperature of 240 F and a pressure of 100 psig. More thorough investigation into the conditions of field failure revealed, however, that because of the complexity of the particular process, the exact conditions were unknown. Accordingly, corrosion testing was first carried out on as-welded samples made from the same material used in the construction of the pipe liner (Ingot CP-2). Environments used were a 15 weight percent solution of hydrochloric acid at 228 F (boiling point), 240 F and 350 F, a 25 percent solution of hydrochloric acid at 284 F and a 33 percent hydrochloric acid solution at 300 F.

Corrosion testing was carried out on welded samples 2 inch by ½ inch samples 0.065 inch in thickness. Following welding in an argon atmosphere, surfaces were ground to a 120 grit surface finish prior to testing. Since the purpose of corrosion testing was only to determine the presence or absence of the localized attack adjacent to welds, corrosion results were evaluated visually. In certain tests quantitative weight loss data were obtained, however, and these reported in mils per year (mpy).

Boiling tests were carried out under atmospheric reflux conditions. The tests conducted at elevated temperatures were made using two liter glassed steel pressure vessels (autoclaves). This made possible a solution volume-sample surface area ratio of 125 cc per square inch, a sufficiently large value to insure establishing valid rates.

Results and Discussion

Figure 1 shows the nature of the selective corrosion attack of the welded zirconium pipe liner. The pipe interior is shown at the T-joint formed by the

TABLE 1—Chemical Analysis of Zirconium Materials

Element	Commercially Pure (Ingot CP-2), ppm,	High Purity (Ingot 2984), ppm
Al	107	< 20
C	400	<300
Co*	< 20	< 20
Cr*	170	48
Cu*	64	< 20
Fe*	1200	200
H**	12**	12
Mg	< 20	< 20
Mn	65	< 20
N	50	42
Ni*	< 20	< 20
Pb	< 20	< 20
Si	70	< 30
Ti	< 20	< 20
V*	< 20	< 20
0	1380	580
Hf	20,000	126
Zr	Balance	Balance

* These elements tend to stabilize the beta form of zirconium.

** Hydrogen contents for the commercially pure material were studied at 12, 4 and 81 ppm.

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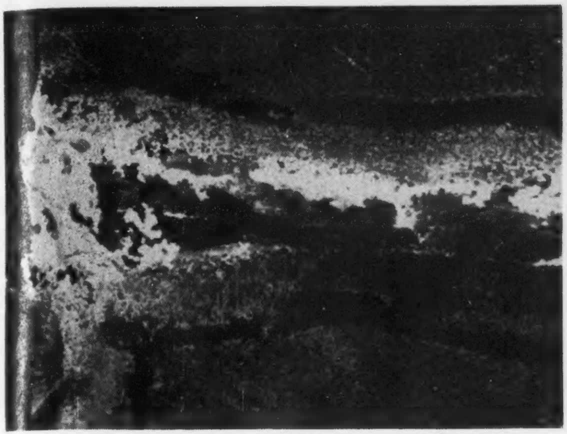

Figure 1—Localized corrosion adjacent to weld in a fabricated zirconium pipe section. Unetched, 3X.

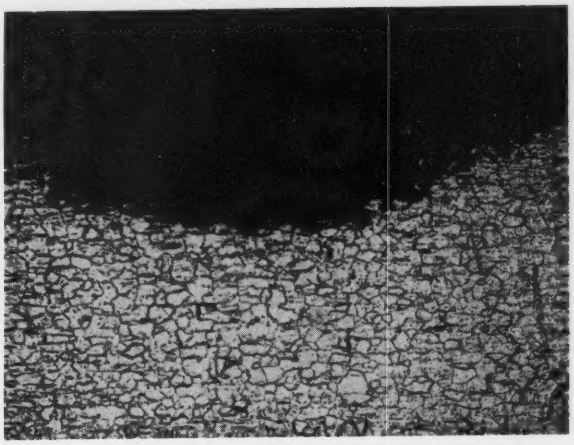

Figure 2—Microstructure present adjacent to zirconium weld in fabricated pipe section. "Transition zone" structure is site of intense attack. $HF-HNO_3-H_2O$ etch-polish, 40X.

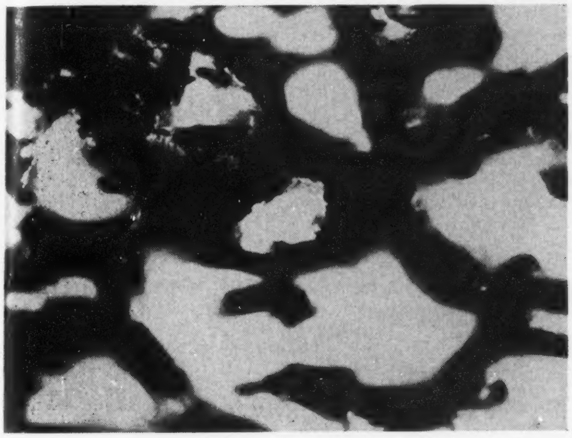

Figure 3—Intergranular trenching attack in transition zone of fabricated pipe section. $HF-HNO_3-H_2O$ etch-polish, 500X.

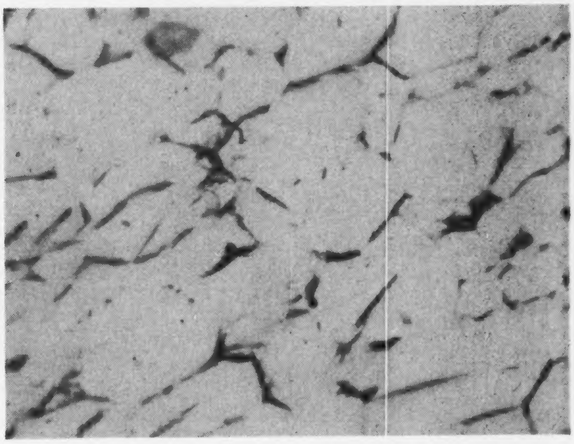

Figure 4—Transition zone microstructure. Intergranular precipitate present to greater extent than in parent metal. $HF-HNO_3-H_2O$ etch-polish, 500X.

pipe-to-flange weld. The appearance of the attack is remarkably similar to that obtained for as-welded Type 304 stainless steel after exposure to the Huey test. In the case of Type 304 stainless steel the attack is associated with chromium depletion at grain boundaries. The attack is characterized by a particular microstructure which exhibits chromium carbide precipitation at grain boundaries with consequent loss of chromium in the adjacent solid solution. The occurrence of this so-called sensitized structure in Type 304 is a result of the material adjacent to the weld having been heated to within a specific temperature range (1000-1400 F) during the welding operation.

A somewhat similar mechanism proved to be responsible for the attack in the zirconium tubing. Figure 2 shows the cross section microstructure at low magnification. To the right of the photograph the typical Widmanstatten structure of the weld is present and on the left the equiaxed grain structure of the parent metal. In the "transition zone" between these two microstructures, intergranular corrosion has occurred. In areas other than these transition zones virtually no corrosion of the zirconium occurred. Figure 3 shows the manner in which trenching occurred around individual grains.

The particular microstructure associated with the transition zone and with the occurrence of intergranular attack is shown in Figure 4. The equiaxed grains are nearly completely enveloped by a precipitate. In the parent material this precipitate is present to a much lesser extent and is discontinuous.

In order to duplicate the type of attack exhibited on the pipe liner, welded samples of the same commercially pure zirconium from which the pipe section had been made (Ingot CP-2, see Table 1) were made and exposed to several hydrochloric acid environments. An environment of 33 percent hydrochloric acid at 300 F was found to cause duplication of the field failure.

A welded sample which was exposed to this environment is shown in Figure 5. The same localized attack is evident that was shown in Figure 1. The microstructure of this test sample is shown in Figure 6. Once again, the association of the transition zone with intergranular attack is evident. Further study of this sample showed that trenching around the grains had occurred in a manner similar to that shown in Figure 3 and that the microstructure was the same as that shown in Figure 4.

The microstructure of normal as-received zirconium of commercial purity is shown in Figure 7. It is composed of equiaxed grains of alpha zirconium with some grain boundary precipitate present. The amount of precipitate present depends upon prior metallurgical history.

A history typical of the CP-2 material involves hot rolling of the cast ingot in the range 1750 to 1600 F. After cleaning, further "hot" rolling in the range 1550 F to 1100 F results in some cold work which is relieved by heat treatment at 1450 F. This rolling and heat treat cycle may be repeated, but the as-received condition is that resulting from the 1450 F heat treatment.

It has been suggested[7, 8] that the grain boundary precipitate may consist of intermetallics of iron, nickel and chromium with zirconium which have been sep-

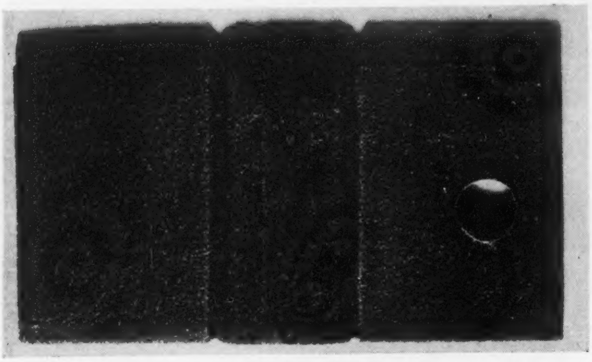

Figure 5—Welded commercially pure zirconium (Ingot CP-2) after 248 hours exposure to 33 percent HCl at 300 F. Unetched, 2X.

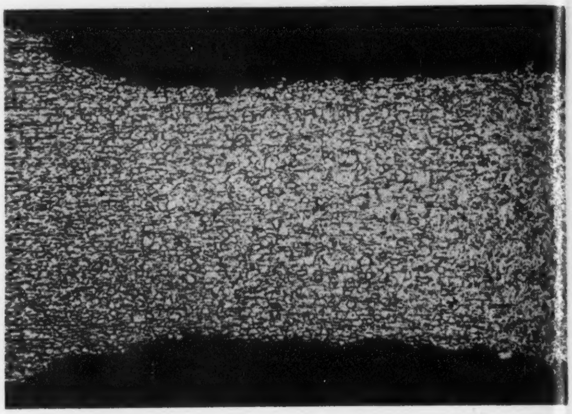

Figure 6—Welded commercially pure sample exhibiting transition zone and associated attack. HF-HNO$_3$-H$_2$O etch-polish, 40X.

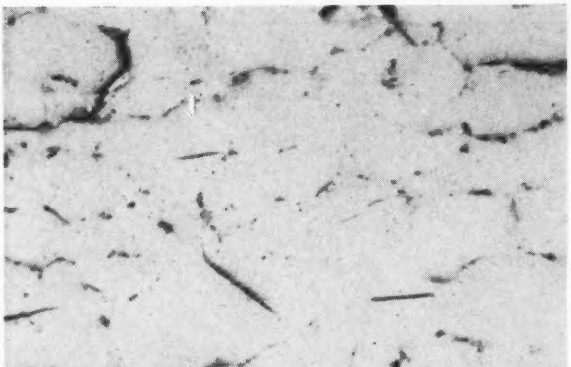

Figure 7—As-received commercially pure zirconium (Ingot CP-2). HF-HNO$_3$-H$_2$O etch-polish, 500X.

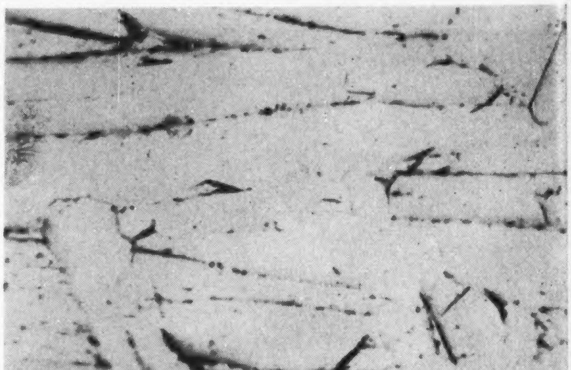

Figure 8—Commercially pure zirconium (Ingot CP-2) after 1800 F and slow cool heat treatment. HF-HNO$_3$-H$_2$O etch-polish, 500X.

arated, during the late stages of cooling through the alpha-beta region, from beta containing high amounts of these alloying elements. Iron, nickel, chromium and other elements noted in Table 1 stabilize the beta form of zirconium and are much more soluble in beta zirconium than in alpha zirconium. The precipitate has also been ascribed to the presence of zirconium hydride.

The transition zone microstructure shown in Figure 4 had been shown to be associated with the selective corrosion in the pipe section and in the test weldments, both of which were made from the same material (Ingot CP-2). It was hypothesized that the increased amount of grain boundary precipitate in this zone was responsible for the attack. The occurrence of the precipitate was at first thought to be associated with the hydrogen content of the zirconium, the precipitate resulting from hydrides. Accordingly, three different hydrogen levels were established in the commercially pure material (Ingot CP-2) by hydrogen charging and vacuum annealing of the 12 ppm hydrogen as-received material. Hydrogen contents of 81 ppm and 4 ppm were obtained in this manner.

During a welding operation the parent metal adjacent to the weld bead is subjected to a bewildering array of temperatures, times at temperatures and cooling rates. In order to isolate the heat treatment causing the excessive grain boundary precipitation and consequent intergranular corrosion and to evaluate the effects of hydrogen content, a series of heat treatments was carried out at temperatures likely to be present during welding. Samples were heated at 1500, 1600, 1700, 1800, and 1900 F for 5, 10, 20, 30 and 40 minutes at temperature followed by water quenching or slow (10°F/minute) cooling and the microstructures of these samples evaluated. This experimental procedure did not reveal any effects which could be ascribed to hydrogen content, but rather specific microstructures were noted for all samples associated with certain heat treatments.

It was found that samples which had been heated to well within the beta zirconium region (to 1800 F) and subsequently slow cooled through the alpha-beta region exhibited increased amounts of grain boundary precipitate in the microstructure. The microstructure typical of a sample of this type is shown in Figure 8. On the other hand, samples of the same material heated in the beta zirconium region (at 1800 F) and then quenched in water exhibited far less grain boundary precipitate. The microstructure typical of this heat treatment is shown in Figure 9.

Although the phase relationships, transformation temperatures and effects of impurities upon the alpha-beta transformation are as yet poorly defined, the work of Picklesimmer[7,8] and others[9,10] suggests a reason for the behavior described previously. It is known that in commercially pure zirconium relatively large amounts of the beta zirconium stabilizers nickel, chromium, manganese and iron are present. These elements have far greater solubility in beta zirconium than in the alpha forms. As zirconium is cooled from 1800 F into the alpha-beta region the structure changes from wholly beta to primary alpha grains plus untransformed beta containing beta stabilizing elements. The untransformed beta is located at the alpha grain boundaries. Further cooling into the alpha region causes the "enriched" beta zirconium to transform into alpha zirco-

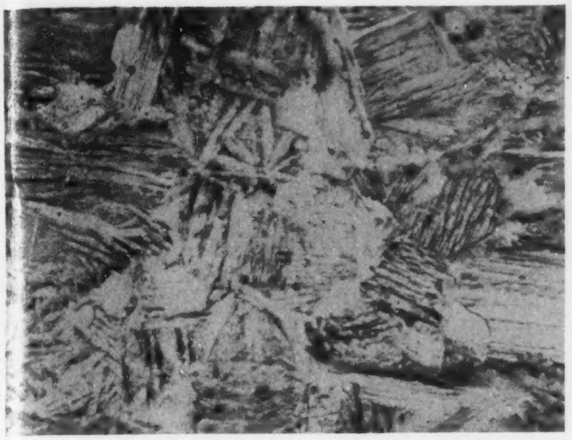

Figure 9—Commercially pure zirconium (Ingot CP-2) after 1800 F and water quench heat treatment. HF-HNO$_3$-H$_2$O etch-polish, 500X.

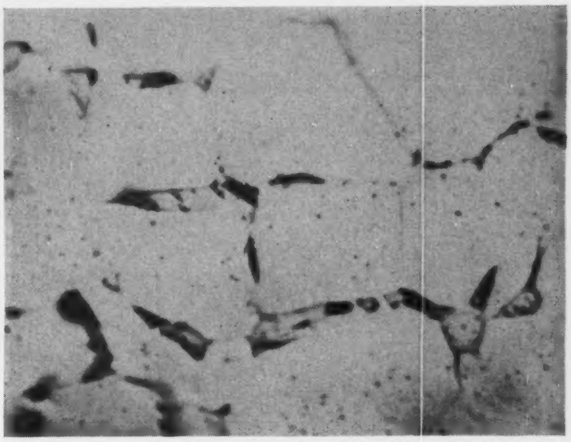

Figure 10—High purity zirconium (Ingot 2984) after 1800 F and slow cool heat treatment. HF-HNO$_3$-H$_2$O etch-polish, 500X.

nium plus intermetallic compounds of zirconium and the beta stabilizers. The alpha zirconium formed during the transition from the alpha-beta region to the alpha region is deposited on the primary alpha grains previously formed leaving the intermetallics located at the grain boundaries. The degree to which this mechanism operates is a function of the mill production procedures but such a mechanism seems to account for the small amount of intergranular precipitate present in as-received material.

The structure of the transition zone of the as-welded samples may be explained using the basic ideas expressed above. The welding operation raises the temperature of the parent metal adjacent to the weld into the alpha-beta region. In this transition zone equiaxed alpha zirconium is transformed into a structure consisting of perhaps 90 percent alpha zirconium and 10 percent beta zirconium. Through the influence of diffusion and preferential solubility the beta zirconium present accumulates increased amounts of the highly soluble stabilizing elements nickel, chromium, manganese and iron. When the weldment is cooled, beta zirconium present in the grain boundary transforms to alpha zirconium and intermetallics heaving an increased amount of the latter in the grain boundaries.

The validity of this explanation of the problem was examined by repeating the heat treating experiment using a high purity zirconium supplied through the cooperation of the Carborundum Metals Co. The chemical analysis of this material is presented in Table 1. As before, samples were heated at 1800 F and either quenched in water or slow cooled. The microstructure typical of a slow cooled sample is shown in Figure 10 while that of a water quenched sample is shown in Figure 11. The slow cooled microstructure exhibits some grain boundary precipitate but it is discontinuous and is present in relatively small amount. The water quenched structure shows practically no grain boundary precipitate. The structural differences between the slow cooled or water quenched commercially pure zirconium (Figures 8 and 9) and the slow cooled or water quenched high purity zirconium (Figures 10 and 11) are very striking. The presence of the Widmanstatten structure in Figures 8 and 9 is generally ascribed to the higher oxygen content of the commercially pure material.[11]

In order to establish more firmly the validity of the concepts developed another experiment was undertaken. Samples of the high purity zirconium were welded and corrosion tested (in the as-welded condition) in the same environment as that which had produced the localized intergranular corrosion in the as-welded commercially pure material. The result of this test, carried out in 33 percent hydrochloric acid solution at 300 F is shown in Figure 12. The sample, after having been tested for five 48 hour periods, exhibited no evidence of selective attack.

Finally, to evaluate the anticipated influence of heat treatment upon eliminating the selective attack, welded samples of commercially pure zirconium were prepared and received the heat treatment of 40 minutes at 1800 F followed by water quenching. These samples were corrosion tested in the 300 F 33 percent hydrochloric acid in comparison with as-welded samples of the same material. Following five 48 hour test periods the appearance of the samples was shown in Figure 13. The 1800 F water quench heat treatment completely eliminated the localized corrosion.

The microstructures of the slow cooled CP-2 samples and of the "transition zone" in the welded samples do not correspond exactly. Their similarity is in the presence of large amounts of precipitate. The reason for this, it is believed, is the difficulty of reproducing exactly in a massive sample the transition zone microstructure observed in the as-welded zirconium. Careful, extensive investigation of the alpha-beta region and the effects of various impurities on the alpha-beta transformation will be required in order to resolve this problem completely. In spite of the lack of exact correspondence of microstructure, however, the direct evidence of the as-welded versus the welded and heat treated materials is sufficient to establish the validity of the assumed hypothesis. There are two possible explanations for the intergranular attack itself. The attack may be the result of a galvanic relationship between the intermetallic compounds precipitated at the grain boundaries and the alpha zirconium immediately adjacent to the grain boundaries. Electrode potentials have not been established for these constituents, however, so that the validity of a galvanic mechanism is open to question.

Another explanation for the intergranular attack may be suggested. Evidence exists that beta zirconium, existing in grain boundary areas in the lower alpha-beta region, transforms on cooling to alpha zirconium and intermetallic compounds of zirconium with iron, chromium,, etc. It seems possible that if cooling occurred quickly the grain boundary beta zirconium, in which is segregated large amounts of the beta stabilizing elements, might transform to alpha zirconium containing the stabilizing elements in metastable solid solution. If this were true, the intergranular attack could be a function of the cleanliness of the zirconium, the "contaminated" zirconium at the grain boundary being attacked to a greater extent than the relatively clean material within the grains. A specific temperature range of hydrochloric acid service would probably be required for this selective intergranular attack since at high temperatures (350 F) both "clean" and "contaminated" zirconium would exhibit high rates while at temperatures lower than perhaps 280 F, rates would be low for both conditions. At intermediate temperatures, excessive attack of the contaminated zirconium could occur while the clean material remained unaffected.

It should not be concluded from this account that welded zirconium is unsuitable as a material of construction for hydrochloric acid services. It is highly satisfactory in many such services and

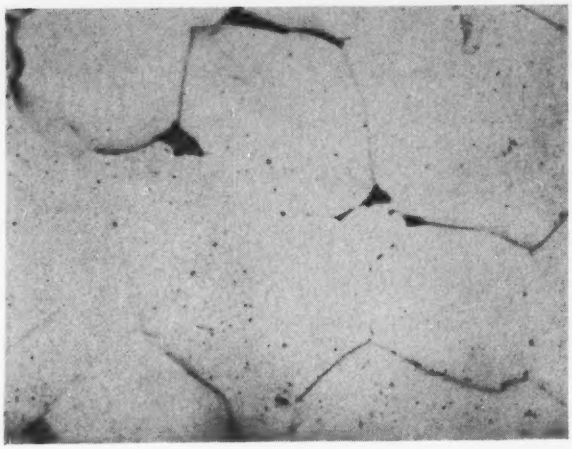

Figure 11—High purity zirconium (Ingot 2984) after 1800 F and water quench heat treatment. $HF-HNO_3-H_2O$ etch-polish, 500X.

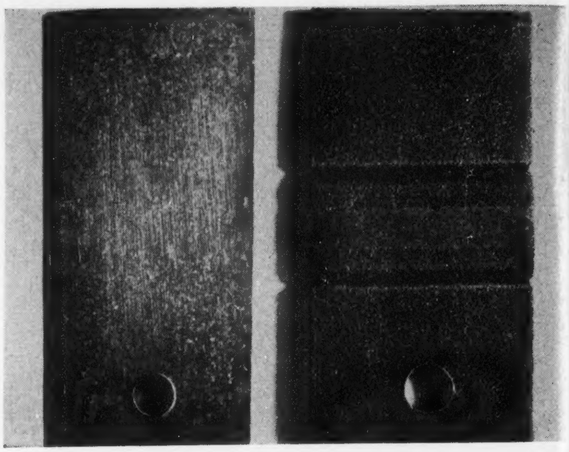

Figure 13—Welded commercially pure zirconium (Ingot CP-2) after 248 hours exposure to 33 percent HCl at 300 F. Sample on left welded and heat treated at 1800 F plus water quench. Sample on right as-welded. Unetched, 2X.

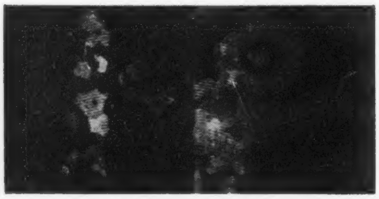

Figure 12—Welded high purity zirconium (Ingot 2984) after 248 hours exposure to 33 percent HCl at 300 F. Unetched, 2X.

is one of the few metals resistant to these environments. It is reasonable to suggest, however, on the basis of the field experience and experimental work reported here that in very severe hydrochloric acid environments certain precautions be taken with welded zirconium.

Remedial Measures

Of outstanding importance in this investigation was the effect of zirconium purity upon the occurrence of the localized type of attack. As-welded high purity material exhibited no trace of the transition zone microstructure or the localized attack adjacent to the weld as did the commercially pure material. It is evident, then, that one method for avoiding this type of attack is to use high purity zirconium.

A second method of preventing the localized attack adjacent to welded commercially pure zirconium is post welding heat treatment. Heating into the beta region at 1800 F for 40 minutes followed by a water quench eliminates the localized attack. Much remains to be discovered about the complex metallurgy of zirconium however, and this heat treatment is not necessarily an optimum one.

Future Work

In the present work the phenomenon of localized attack in HCl adjacent to zirconium welds has only been described and the outlines of the problem sketched. Work is now in progress designed to establish reliable corrosion rates for the several metallurgical structures obtained by a series of heat treatments. This program may establish more exactly what is taking place in the transition zone during welding.

Other heats of presently available commercial purity material are to be evaluated and a more accurate heat treatment established for correction of the weld-induced localized failure.

Summary

A localized type of corrosion adjacent to welds in a commercially pure zirconium was determined to be associated with the presence of a so-called "transition zone" microstructure.

The transition zone microstructure consists of equiaxed alpha zirconium grains surrounded by a grain boundary precipitate present to a far greater extent than in the parent material. This microstructure is produced by the welding operation which brings about a combination of heating into and slow cooling through the low alpha-beta region. Under these conditions beta stabilizers and beta phase zirconium will segregate at grain boundaries to be precipitated as alpha zirconium and intermetallics upon further cooling to the alpha region.

Experimental heat treatments and subsequent corrosion testing of massive (unwelded) zirconium samples demonstrated that water quenching from a temperature well within the beta zirconium region resulted in reduced corrosion as compared to material slow cooled from this temperature (through the critical alpha-beta region).

The use of high purity zirconium in as-welded samples resulted in the elimination of localized attack in the as-welded material.

The intergranular attack suffered in the transition zone microstructure was ascribed to a galvanic mechanism involving alpha zirconium and the grain boundary precipitate.

It is concluded that remedial measures to be taken to prevent the "weld decay" type of attack would include (1) use of high purity zirconium and (2) heating of commercially pure zirconium weldments at 1800 F followed by a water quench.

DISCUSSION

Question by Otto H. Fenner, Monsanto Chemical Co., St. Louis, Mo.:

What value do you find occurs through the employment of a water quench welding technique in the fabrication of equipment of zirconium?

Reply by B. S. Payne:

It is conceivable that a water quench welding technique would result in a lessened precipitate in the grain boundaries at the edge of the Widmanstatten structure. One of the most important problems in welding zirconium is to insure that interstitial gases do not come in contact with metal that is above approximately 1000 F. If water were used as an immediate quench there would undoubtedly be an impurity pick up in the weld, thus greatly decreasing ductilty and, to a lesser extent, reducing the corrosion resistance.

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T-6A-16 Coal Tar Coatings

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T9 Marine Biological Deterioration

T-9A Biological

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T-9B Preservatives and Their Appraisal

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TECHNICAL ARTICLES SCHEDULED FOR PUBLICATION

May Issue

Suggested Mechanisms of Changes With Time in the WP-1 Metal Conditioning Composition, by E. R. Allen

Acid Volume and Inhibitor Quantity—Their Effect on Corrosion of Steel in Hydrochloric Acid, by William E. Billings and David Morris

Corrosion of Nickel Base Alloys in High Temperature Nitrogen Environments, by J. S. Brunhouse and G. W. Titus

Corrosion of Stainless Steels by Hot Hydrogen Sulfide, by F. J. Bruns

Cathodic Polarization of Steel in Various Environments, by A. R. Erben

New Methods of Simulating Corrosive Plant Conditions in Laboratory, by A. O. Fisher

Chemical Inhibition of Mineral Scales, by L. W. Jones

Corrosion of Aluminum Alloys in High Purity Water in 150-340 C Temperature Range, by D. R. MacLennan

Aluminized Steel Wire, by Jane Rigo

Growth of Single Iron Crystals for Corrosion Studies, by Willard F. Brickell and Edward C. Greco

June Issue

Effect of Sunlight on Corrosion of Steel, by Leonard C. Rowe

Use of Boehmite Films for Corrosion Protection of Aluminum, by D. Altenpohl

Acid Corrosion Inhibition With Secondary Acetylenic Alcohols, by John C. Funkhouser

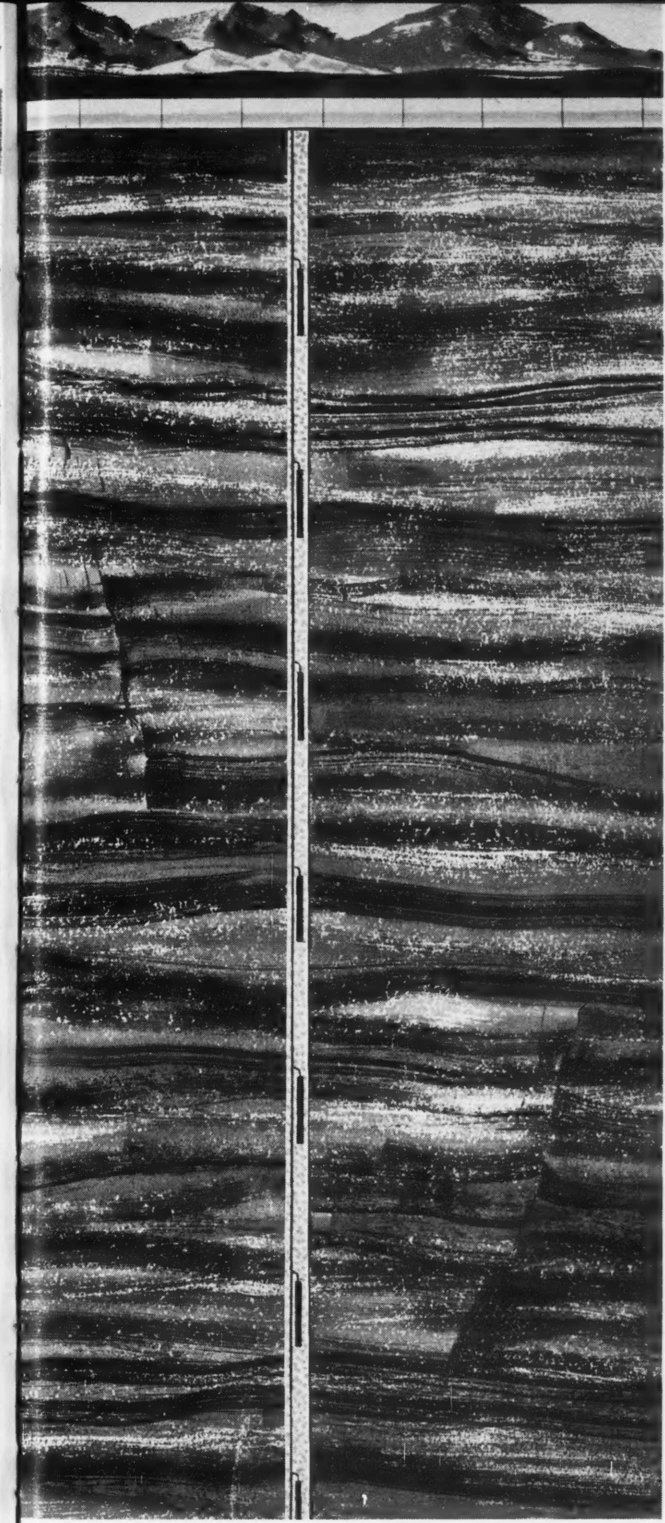
Corrosion of Superalloys at High Temperatures in Presence of Contaminating Salts, by A. Moskowitz and L. Redmerski

Devices Used in Corrosion Control of Power Cables, by J. J. Pokorny

Unity of Anaerobic and Aerobic Corrosion Process in the Soil, by C. A. H. von Wolzogen Kuhr

Corrosion of Carbon and Low Alloy Steel in Out-of-Pile Boiling Water Reactor Environment, by D. C. Vreeland, G. G. Gaul and W. L. Pearl

Stress Corrosion Cracking: A Review of Current Status, by W. L. Williams



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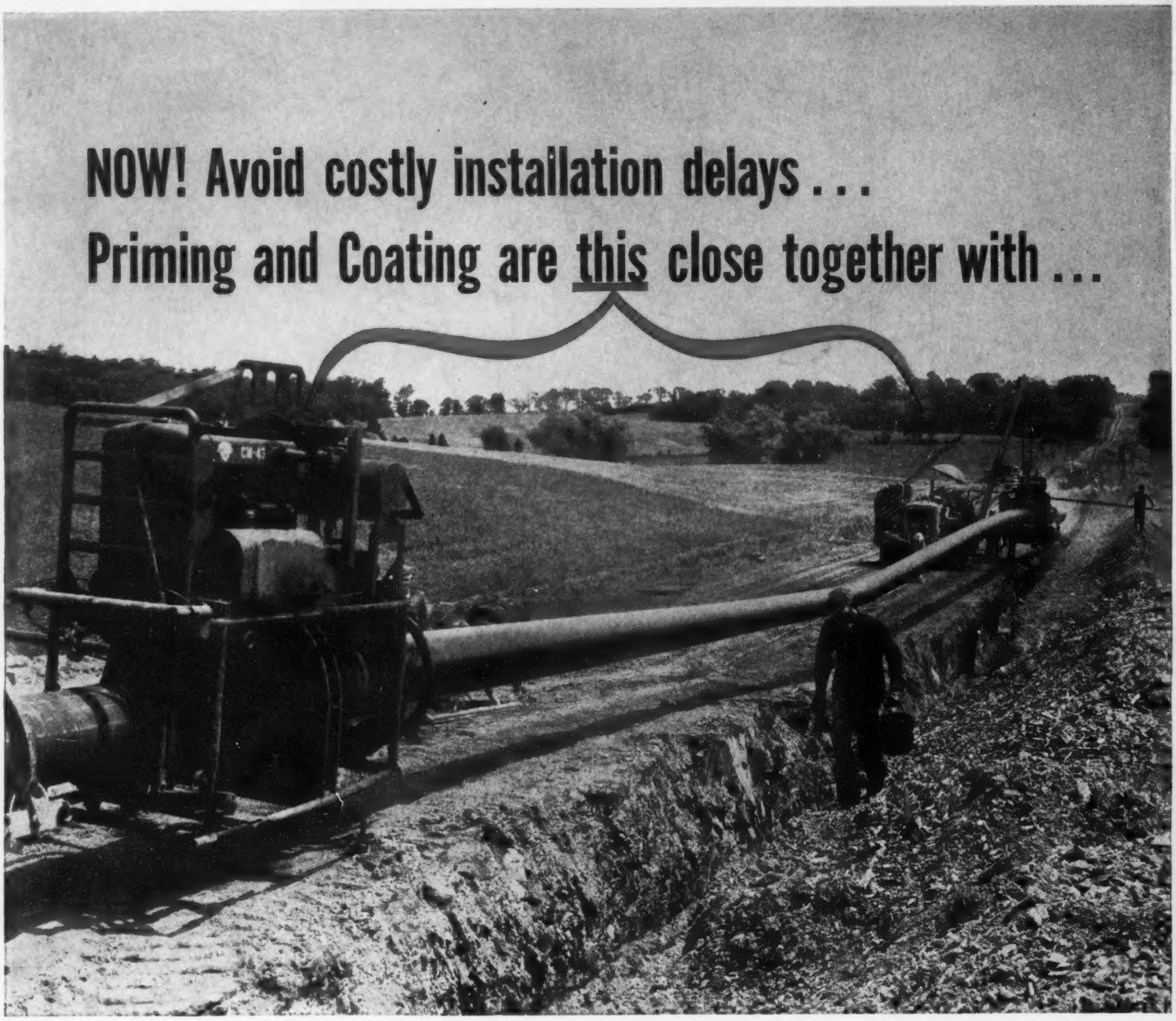

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